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Effect of infrared radiation upon an oleoresinous enamel : compregnation of wood testing of dimethylolurea plastic

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THE EFFECT OF INFRARED RADIATION UPON AN OLEORESINOUS ENAMEL

COMPREGNATION OF WOOD
TESTING OF DIMETHYLOLUREA PLASTIC

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(1) THE EFFECT OF INFRARED RADIATION UPON AN OLEORESINOUS ENAMEL

(2) COMPREGNATION OF WOOD
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ACKNOWLEDGMENT

The Author wishes to acknowledge the kind assistance and helpful guidance of Doctor R. C. Ernst, who directed this research.

ABSTRACT

The purpose of this investigation is to determine the effect of infrared radiation on the structure of an oleoresinous enamel made of Linseed Oil, a Bakelite, Resin, and Titanium Dioxide as the pigment.

Enamels were prepared by using various ratios of titanium dioxide and lampblack as the pigments to give different degrees of infrared reflectance. The resulting enamels were cast into films which were weathered. The aged films were then tested for distensibility and finally extracted with acetone.

It was concluded that between 40% and 60% reflectance of the infrared a maximum polymer content is obtained. It is postulated that this may be caused by a combination of (1) a retardation of oxidation by the pigments used, (2) a breaking down of polymers already formed, or (3) the failure of a film to receive enough radiation of a specific type to cause a maximum polymer formation.

INTRODUCTION

This investigation has for its purpose to determine the effect of infrared radiation on the polymerization of pigmented oleoresinous varnishes. A Linseed oil - Bakelite type varnish, pigmented with titanium dioxide and lampblack - will be studied.

The character of oleoresinous films has been under investigation by Ernst and others (3, 4, 5) for several years in this laboratory. The previous investigations have established that by the use of an acetone extraction an oleoresinous film may be separated into two constituents: 'polymer' and 'non-polymer'. The polymers are made up of three dimensional polymeric (polymer) substances which serve to form the film and are acetone insoluble. The non-polymerized or slightly polymerized material (non-polymer) are acetone soluble and form the plasticizer portion of the film. These investigations have been concerned with this polymer to non-polymer ratio in connection with the age of the film.

The previous investigators found that during the early life of the film its polymer content increased rapidly but later increased less rapidly until finally a constant value was approached as the film neared failure. It was also found from the investigation of enamels that with the pigments investigated they affected the rate of polymerization only to a

slight extent. The polymer content at failure when the resin-pigment ratio was less than 1-1 depended upon the pigment, the drying oil, and the oil length. When fifty percent of the resin is replaced by pigment in varnishes of the insoluble type resins (Bakelite) the substitution may be made without effectively changing the polymer content at failure. In the soluble type resins (Ester Gum) only inert pigments such as titanium dioxide may be substituted without increasing the polymer content at failure.

It is the purpose of this investigation to determine how radiation in the red and infrared of the spectrum will affect the rate of polymerization and the life of the film. It is hoped that this investigation will further aid in the formulation of satisfactory finishes.

HISTORICAL

It is a well known fact that light of various wave lengths has different effects upon oil and paint films. Considerable work (13,14,15) has been done in regard to the effect of ultra-violet light upon oils and paint films.

The effect of light of longer wave lengths (above 7000 Å) has received little attention in regard to its effect upon paint and enamel films. Greenbank and Holm (1) state that samples of cottonseed oil, butter oil, corn oil, and lard absorb approximately the same amount, about 5 percent, of light between 5400 Å and 7500 Å. They also indicate that the percent of light absorbed increases with decrease in wave length.

Ganguli (6) shows by numerous examples that infrared radiation is effective in only a few cases in bringing about the rupture of molecules. His treatment is largely mathematical and deals with unimolecular reactions.

Considerable work, as previously reported (3,4,5) has been done on the change of polymer content in the aging of films. The initial investigation in these laboratories was conducted by Ernst and McQuilkin (3), who worked with varnish films prepared by cooking Congo Resin with bodied Linseed oil and calorized Tung oil.

Various oil lengths were used, and the enamels were prepared by grinding titanium dioxide in these varnishes. The change in polymer content and the distensibility change were studied throughout the life of the film. Ernst and McQuilkin observed that the concentration of polymer, as indicated by acetone extraction, reached a value of from 70% to 80% at failure of the film, as indicated by minimum distensibility. From these observations they concluded that during the life of an oleoresinous film a complicated network of three-dimensional oil and resin molecules is formed which gradually decreases the elasticity of the film until failure occurs.

Ernst and Lubbers (4) extended this fundamental research so that it included varnishes formulated from typical drying oils and several synthetic resins. The particular resins were chosen because of their variation in reactivity and chemical type. From their observations, using the same experimental methods and modes of correlation as used by Ernst and McQuilkin (3) they concluded that the varnishes could be divided into two classes according to the resin contained. The first class contained resins of the "insoluble" type and tended to promote polymerization, the second class was composed of the "soluble" type resins and tended to retard polymer-

ization. The type "soluble" or "insoluble" is referred to their solubility in acetone.

In continuing the investigation, Ernst and Eudaley (5) worked with enamels in which they varied oil length, type of oil, type of resin, and type and amount of pigment. They concluded, first, that the pigments investigated affected the rate of polymerization only slightly; second, that the polymer content at failure either increased or decreased depending upon the resin, pigment, and oil; third, the magnitude of this variation that occurs is dependent upon the drying oil and the oil length when pigment is substituted for the resin in amounts less than fifty percent; and fourth, where fifty percent of the resin is replaced by pigment, substitution can be made in insoluble type resins with effectively no change; but in soluble type resins only inerts, like titanium dioxide, may be substituted without effect.

THEORETICAL

This investigation does not propose to collect additional data which will further insight into the mechanism of drying of oleoresinous films but attempts to explain the change in films when exposed to light of long wave length. When correlated with previous work, the data obtained in this investigation should be of value in formulating protective coatings.

The mechanism of the 'drying' reaction of a drying oil is generally considered to be a combination of oxygen absorption and molecular association or polymerisation. Although the formation of a film is dependent upon ox ygen absorption, the film does not immediately absorb oxygen but goes through an induction period the length, of which is governed by traces of antioxidants in the oil. The addition of metallic driers appreciably shortens this initial induction period. Most investigators (10, 11, 12) consider this oxygen absorption to form peroxide linkages at the double bonds.

There are many controversial data and opinions concerning the exact nature of the interlinking to form polymers. The first is the combination of unsaturated molecules by primary chemical linkages to form larger molecules, true polymerization. The second type of

polymerisation is that in which combination of unsaturated compounds take place through secondary or residual valencies. This type of polymerisation is called colloidal association.

Powers, Overholt, and Elm (2) list five steps as discernible in the drying and aging of a film: 1) a period of induction before oxidation is appreciable; 2) a period of most rapid oxidation; 3) oxygen apparently rearranges, forming conjugated systems if they are not already present; 4) polymerization accompanied by a rapid increase in density, a rapid decrease in specific refraction, and a rapid drop in iodine value; and 5) the aging step occurring more slowly and characterized by depolymerization or oxidative decomposition of the polymers previously formed.

Rhodes and Goldsmith (9) state that Lampblack and Carbon black markedly inhibit the oxidation of Linseed oil. On the basis of oxygen absorption, enamels containing either of these pigments should contain less polymer at a definite age than either clear varnishes or other enamels in a linseed oil vehicle. In their experiments, however, they used a large ratio of pigment to oil, i.e., one part of pigment to nine parts of oil by weight.

Investigations by Greenback and Holm (1) point

to the fact that the percent absorption of light and the peroxide formation increases with decreasing wave length of the light. Ganduli (6) postulates that it is possible for infrared radiation to rupture a few types of molecules. On the basis of these data it is indicated that the polymers would be slow to form and after formation may be broken either by the action of the infrared or the depolymerization or oxidative decomposition during the aging period.

The previous investigations (3,4,5,) have revealed that the percent elongation of a film when freshly prepared is large and the polymer content is low, but that it decreases and approaches a minimum value as the polymer content reaches its constant value. This diminishing distensibility is a measure of film failure.

EXPERIMENTAL

RAW MATERIALS

A 16 gallon Linseed Oil - Bakelite varnish was used in this investigation. The drying oil used was a Z-3 bodied Linseed oil.

The pigments used were C.P. Titanium Dioxide and Lampblack. Pure titanium dioxide was ground in the oil to form a paste and a second paste was made by grinding titanium dioxide and a small percentage of lampblack with the oil. The ratio of oil to pigment was such that in making the enamels it was necessary only to weigh out a calculated amount of the varnish and the pastes. The varnish and pastes, together with the thinner and drier, were thoroughly mixed to secure a homogeneous enamel.

Xylene was used to thin the enamels to approximately the same percentage solids as the original varnish. Drier was added to the formulations in the form of regular Nuodex lead, manganese, and cobalt drier, containing 24, 6, and 6 percent metal, respectively. They were added to the enamel formulations on a metal to oil-added weight basis of 0.30% lead, 0.03% manganese, and 0.015% cobalt.

The pigment was added on the basis of replacing one half of the resin with pigment. This was to insure good hiding and to duplicate the type of enamel found to be most satisfactory for this investigation after a close examination

of the data of Ernst and Eudaley.

When exposed to ultra-violet light, this enamel gave only a small polymer change during the aging cycles. The light source used emitted bands of ultra-violet in addition to red and infrared, the effect of the ultra-violet would be minimized by the use of the Linseed oil-Bakelite varnish pigmented with titanium dioxide.

APPARATUS

The apparatus used in this investigation consisted chiefly of an accelerated weathering unit, a distensibility tester, a battery of extraction units, and a multipurpose reflectometer. Other equipment included a paint mill, a film caster, a dial gauge micrometer, and a constant-temperature, constant-humidity cabinet. All this equipment is described in the following paragraphs.

ROLLER MILL. A three-roll, differential-speed paint-mill was used to disperse the pigment in oil. Several passes were made to insure complete dispersion.

FILM CASTER. The films for this investigation were cast by means of a doctor blade having a clearance of 0.006 inches. The caster has an overall width of 4½ inches and deposits films 3½ inches wide.

PANELS. All films were cast on 31 gauge coke tin panels 5x10 inches in size, having ¼ inch holes centered ½ inch from the top.

ACCELERATED WEATHERING UNIT. Rapid weathering was brought about by the use of an accelerated-weathering unit, Model X-L, manufactured by the National Carbon Company. The unit is in the form of a cylindrical, copper-coated, vertical steel shell, containing a rotating panel rack. This rack has a carrying

capacity of 64 panels and rotates 360 degrees every 118 minutes. Radiation was produced by the use of "National" E Carbons which have a high emission value in the red and infrared. (17)

CONSTANT-TEMPERATURE, CONSTANT-HUMIDITY CABINET. An insulated box maintained at 70° F. and 54% relative humidity served as a constant-temperature, constant-humidity cabinet. The temperature was maintained by a thermostatically operated incandescent lamp heater. Humidity was controlled by the use of a saturated solution of magnesium nitrate hexahydrate, in an open pan as suggested by Gardner (7). Such a solution, when contained in a compartment such as this and at 70° F., will hold the atmosphere of the enclosure at 54% relative humidity.

FILM CUTTING TEMPLATE. A 6x1x3/8 inch bar of cold-rolled steel was polished on one 1x6 inch face and used as a template for cutting the strips of films used in the elongation tests.

MICROMETER. The thickness of the free enamel films was measured with a Randall & Stickney dial gauge micrometer. The dial is graduated in thousandths of an inch and readings can be interpolated to ten-thousandths of an inch.

EXTRACTION UNITS. After aging and elongating the films were extracted in Sohxlet extraction units. Each unit consisted

of a 400 ml. extraction flask, a siphon cup, an extraction thimble, and a condenser cap. The siphon cup containing the extraction thimble with the film was suspended by fine wires from the condenser cap which rested on the neck of the flask. The units were contained in five batteries of twelve units each. The batteries were in the form of rectangular wooden boxes lined with asbestos paper, and fitted with removable lids in which holes were cut to receive the extraction flasks. The flasks were placed in cylindrical metal reflectors which directed the heat to them from the 75 watt incandescent lamp heaters. Cooling water for the condensers was supplied through an iron pipe manifold and the cooling system of the batteries were connected in series.

REFLECTOMETER. A Hunter (16) multipurpose reflectometer was used to determine the percentage of infrared reflection. A Wratten 87 filter was used. The instrument was standardized for this filter by use of a porcelain standard which had a reflectance of 81.9 percent when compared with Magnesium oxide, which has a reflectance of 100.0 percent.

DISTENSIBILITY TESTER. The distensibility tester used was manufactured by Testing Machines Inc. This machine is equipped to record automatically the elongation and load

under which a film failed. The rate of pull is 4 inches per minute, maximum elongation is 27 mm., and maximum load obtainable is 10 kilograms.

PROCEDURE

The procedure consisted of preparing the enamel, casting the film on panels, aging, and determining the reflectance values at various ages. The panels were then amalgamated and the film stripped free. The free films were conditioned, elongated, and extracted. An explanation of each step follows.

PREPARATION OF SAMPLES. As previously mentioned the pigments were ground in oil so that a definite ratio existed between pigment and oil. It was necessary only to weigh out the varnish and add a definite weight of pastes, driers, and thinner. This was then agitated to obtain a homogeneous mixture. The samples were checked for thorough dispersion of the lampblack by the use of a microscope.

CASTING OF FILMS. Plate glass panels were leveled, a few drops of water spread on their surface, and the tin panels, thoroughly cleaned with xylene, were firmly pressed onto the wetted glass surface. It is necessary to obtain a good adhesion between the glass and the tin panels. The films were then cast with the doctor blade using a constant forward pull of 1-2 inches per second. All samples were cast in duplicate.

WEATHERING. The films were allowed to dry in air until they were tack free. The panels were then trimmed to 3½ by 10 inches and placed in the accelerated weathering unit.

They were exposed to the action of infrared light and water for period of 2, 4, 6, 8, and 10 cycles. A cycle was taken as 12 hours of light and water. This differed from the previously used cycle in that there is no period of solely light or water. No rest or refrigeration period is used. This cycle was chosen because it closely approximated all government specifications as to the conditions of accelerated weathering. The panels were taken from the unit at the end of each even cycle, inspected, and their percent reflectance determined.

REFLECTANCE. The percent reflectance was determined for each film at the end of each even numbered cycle. The Hunter reflectometer is so constructed that a panel will fit conveniently over the aperture for the sample. The machine was standardized against the porcelain standard at the beginning of each set of determinations and the value checked after making the readings.

AMALGATION AND STRIPPING. After removal from the accelerated weathering unit, the panels were inclined at an angle of approximately twenty degrees with the horizontal and a drop of mercury placed on the unprotected metal surface above the top edge of the film. After remaining in this position overnight the tin surface was completely amalgated. This

surface is such that there is only a small degree of adherence with the film so that it is possible to lift one end of the film with the assistance of a razor blade and strip the film from the panel.

CONDITIONING. The free films were hung in a constant-temperature, constant-humidity cabinet for at least three days; at the end of this time the films were considered to reach temperature-humidity equilibrium with the atmosphere of the cabinet.

DISTENSIBILITY TESTING. Two strips measuring 1x6 inches were cut from each conditioned film. The strips were cut with a razor blade, using a steel template as a pattern. The thickness of each film was determined by averaging several readings taken with a dial gauge micrometer. The sample strips were then clamped in the Testing Machine so that the sample length was 100 mm. The clutch of the machine was then engaged and a screw released so that the machine would record the amount of elongation. The percent elongation and load were read directly from the attached scale.

EXTRACTION. The polymer content of the films was determined by extracting with acetone to constant weight and considering the insoluble as polymer. A double thickness of #30 Whatman

filter paper was folded into a double thickness, Whatman extraction thimble. This thimble was then extracted with acetone for two days to permit it to reach constant weight. It was then dried at 105° C for two hours, cooled in a dessicator, and weighed. The weighings were performed in a large weighing bottle on an analytical balance. The film sample was cut into small bits and placed in the thimble. The thimble was again dried, cooled, and weighed, the weight of the sample being determined by difference. The thimble, containing the sample, was then placed in an extraction unit and extracted for approximately two weeks to constant weight. An empty thimble was extracted with each ten samples as a blank to minimize any error resulting from slight changes in the weight of the thimble. The loss in weight of the sample was obtained by difference and the percent of insoluble material remaining was calculated.

RESULTS

The formulations, shown in Table 1, were cast into films, aged, and the elongation, tensile strength and unextractable determined at various ages. It was impossible to obtain carbons for the weathering unit which had an emission spectra high in the red and infrared without considerable radiation in the violet and ultra-violet. To minimise the effect of the shorter wave lengths of light the enamel selected was one that Ernst and Eudaley (5) Fig.1, had shown wherein the polymer content increased very slowly when exposed to ultra-violet radiation. This enamel was made from a 16 gallon Linseed oil-Bakelite varnish with 1/2 of the resin replaced with titanium dioxide.

The results shown in Table 11 were based on the determination of these experimental values at the end of 2,4,6,8, and 10 cycles as well as for unaged films. The percentage reflectance is the average of the values obtained at the end of each even cycle during the aging of the film. The reflectance varied slightly during the aging process but never over 3% from the average. The readings from the Hunter Reflectometer when corrected by consulting the correction curve for the instrument give the percentage reflectance of the film when magnesium oxide is taken as 100 percent reflectance of the infrared.

TABLE 1

Formulations

Code #	Percentage of Pigment		Approx. Percent Infrared Reflectance
	Lampblack	Titanium dio.	
TLB1-5-1	0.00	100.00	81.6
TLB1-5-2	0.05	99.95	63.0
TLB1-5-3	0.10	99.50	55.5
TLB1-5-4	0.25	99.75	43.5
TLB1-5-5	0.75	99.25	27.5
TLB1-5-6	2.41	97.59	14.8

TLB1-5, refers to a Linseed oil -Bakelite varnish, 16 gallon oil length, with one half of the resin replaced with pigment.

TABLE 11

Physical Properties of Enamel Formulations at Varying Ages

Sample	Physical Property	Age in Cycles 0	2	4	6	8	10
TLB1-5-1	% Reflectance	81.6	82.3	80.8	78.6	79.5	78.7
	% Elongation	***	- -	- -	- -	6.66	6.66
	% Unextractable	51.9	54.7	70.6	67.4	69.9	69.2
TLB1-5-2	% Reflectance	63.0	62.2	61.5	61.8	61.0	61.0
	% Elongation	***	27.4	7.2	8.75	4.7	4.9
	% Unextractable	60.5	63.9	70.3	69.5	68.5	72.3
TLB1-5-3	% Reflectance	55.5	55.2	55.0	55.4	54.8	55.0
	% Elongation	***	5.4	4.5	5.4	3.4	4.25
	% Unextractable	58.4	68.9	67.3	72.6	75.0	72.8
TLB1-5-4	% Reflectance	43.7	42.9	42.9	43.0	42.9	43.3
	% Elongation	***	7.7	4.48	4.0	4.56	4.27
	% Unextractable	61.9	69.8	70.1	75.5	74.5	76.7
TLB1-5-5	% Reflectance	27.8	27.6	27.2	27.3	27.3	27.4
	% Elongation	***	6.0	2.7	3.7	3.5	1.5
	% Unextractable	58.7	69.8	66.9	70.00	72.9	70.2
TLB1-5-6	% Reflectance	13.8	15.7	14.9	14.6	14.2	14.3
	% Elongation	***	2.0	2.0	1.8	1.5	1.5
	% Unextractable	51.6	61.1	64.8	60.4	65.0	60.7

*** - Too elastic to elongate
- - -Different testing machine used.

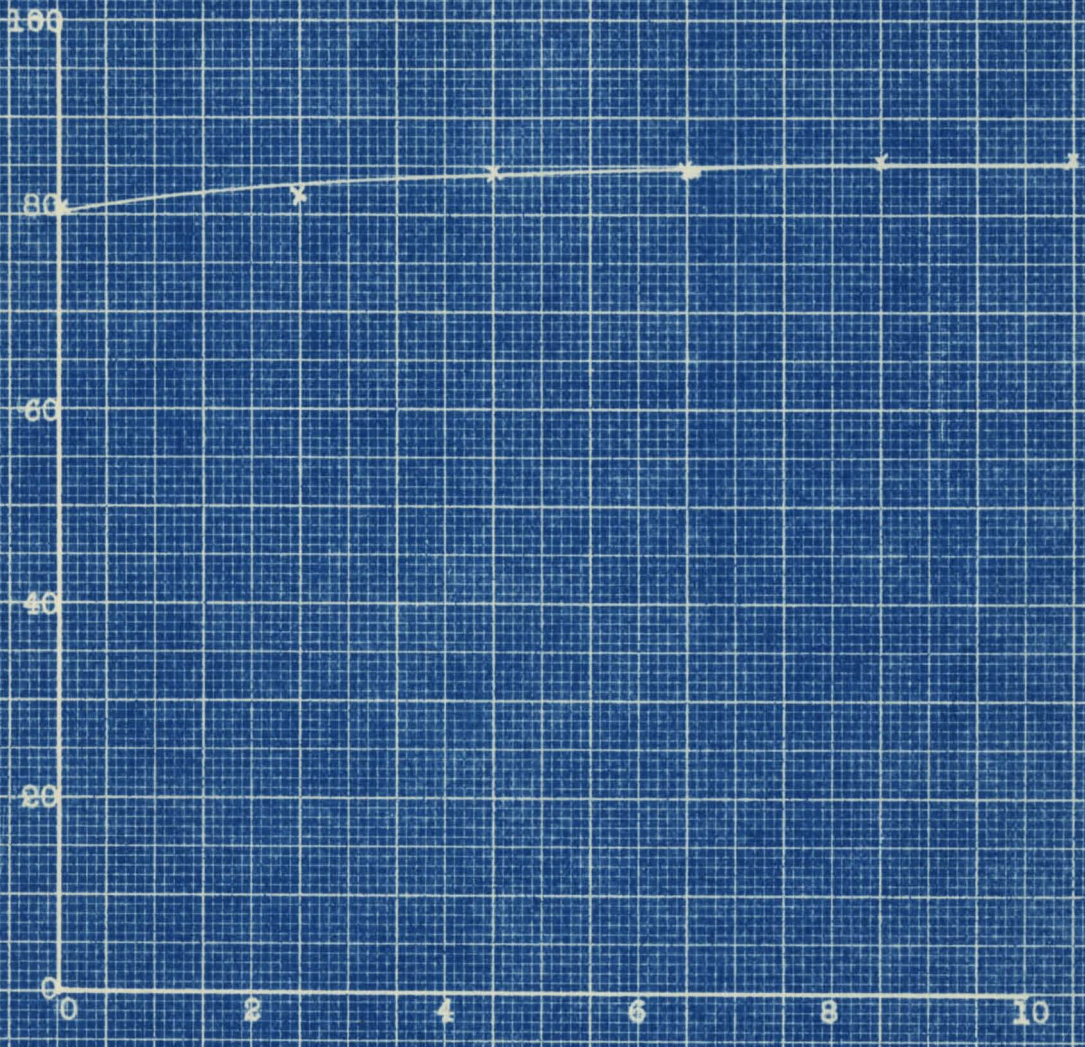

FIG. 1. VOLUME PERCENT UNEXTRACTABLE VS. AGE FOR 16 GALLON LINSEED OIL-BAKELITE VARNISH WITH ONE-HALF THE RESIN REPLACED BY TITANIUM DIOXIDE

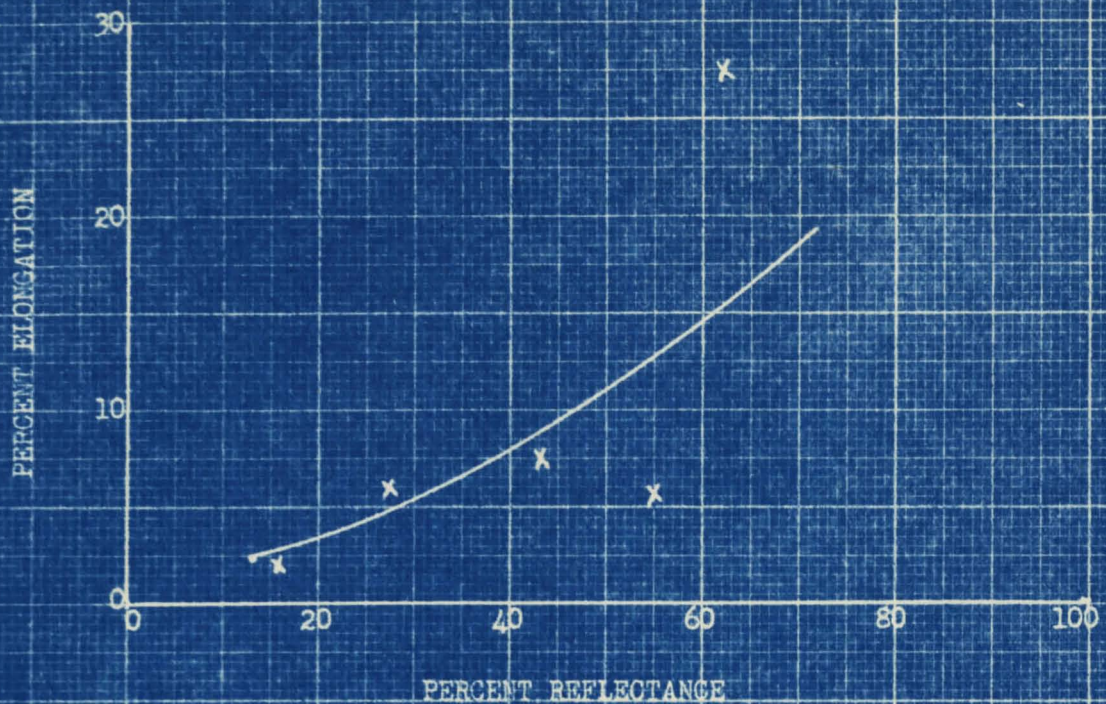

FIG. 2. PERCENT ELONGATION VS. PERCENT REFLECTANCE OF ENAMEL AT 2 CYCLES AGE

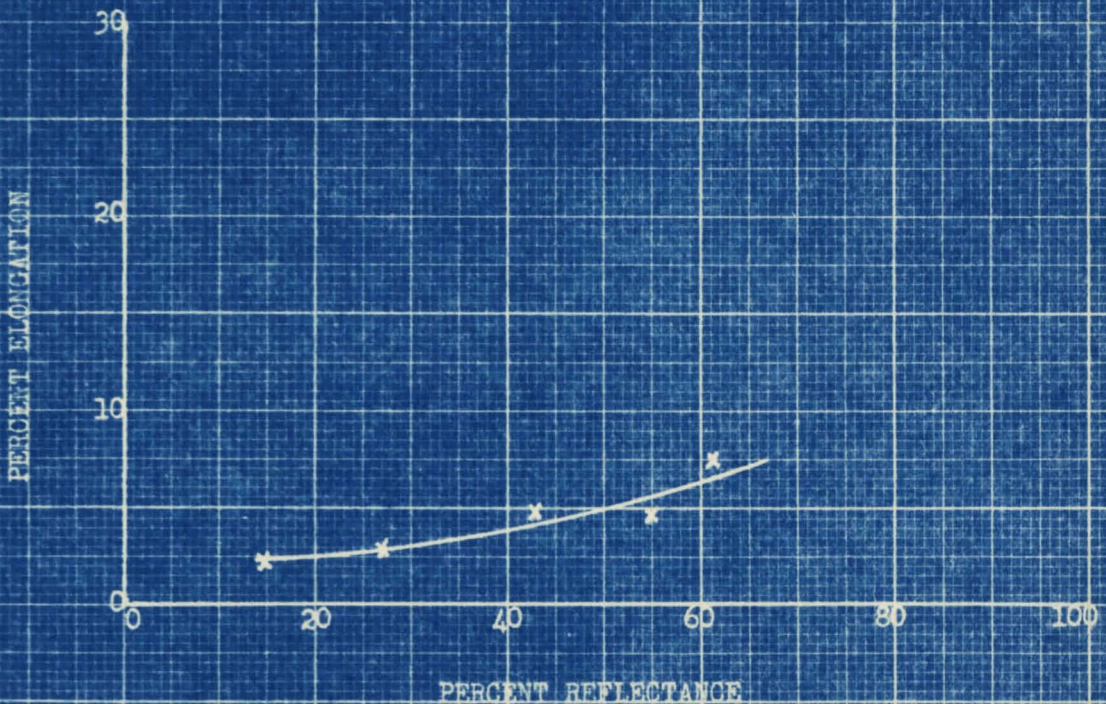

FIG. 3. PERCENT ELONGATION VS. PERCENT REFLECTANCE OF ENAMEL AT 4 CYCLES AGE

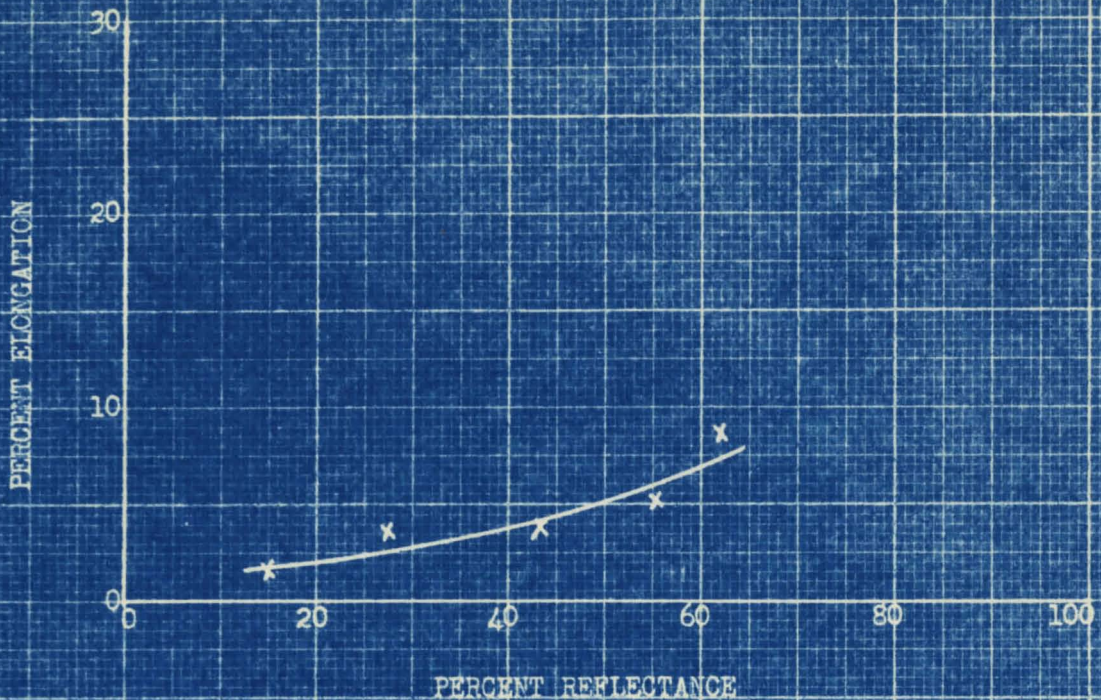

FIG. 4. PERCENT ELONGATION VS. PERCENT REFLECTANCE OF ENAMEL AT 6 CYCLES AGE

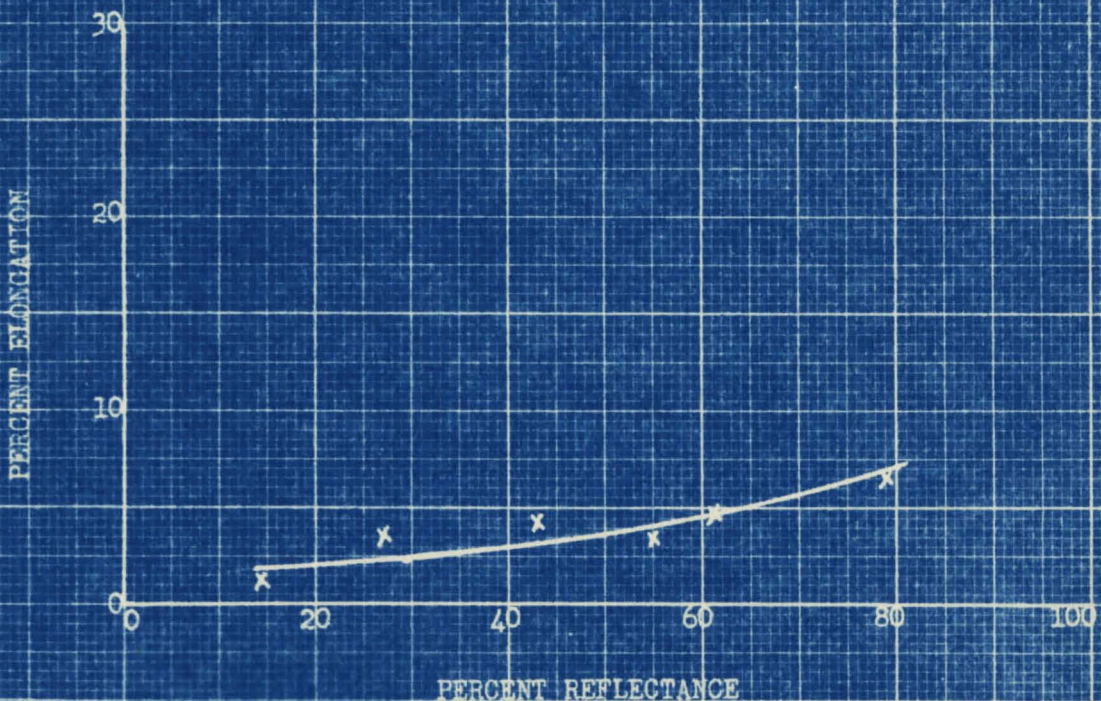

FIG. 5. PERCENT ELONGATION VS. PERCENT REFLECTANCE OF ENAMEL AT 8 CYCLES AGE

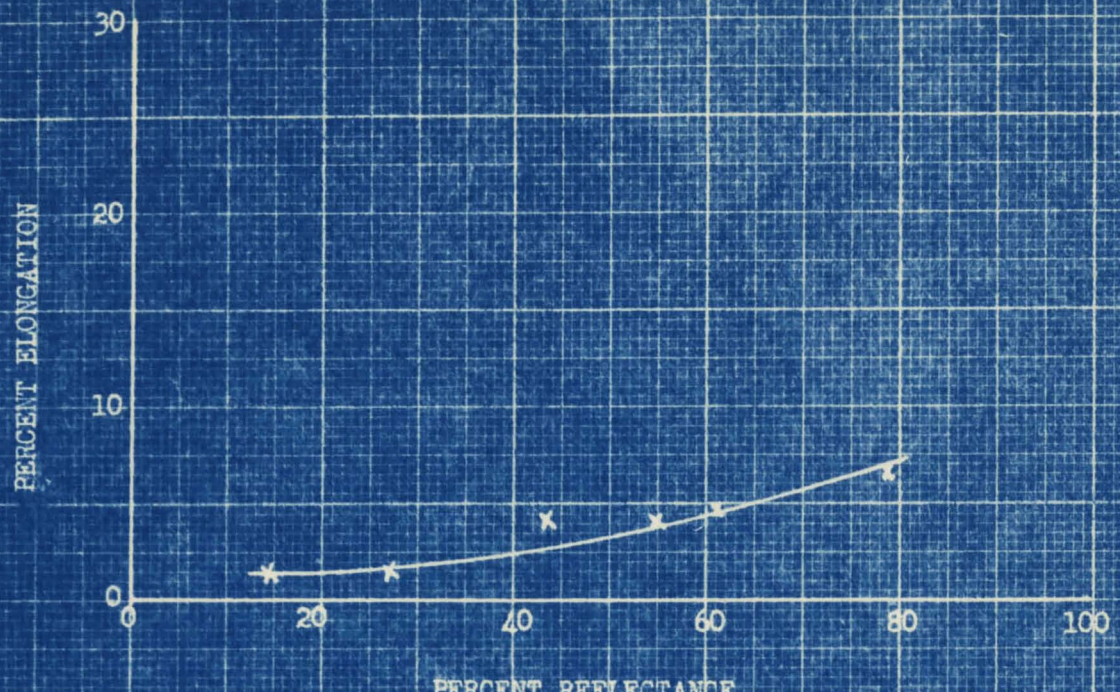

FIG. 6. PERCENT ELONGATION VS. PERCENT REFLECTANCE OF ENAMEL AT 10 CYCLES AGE

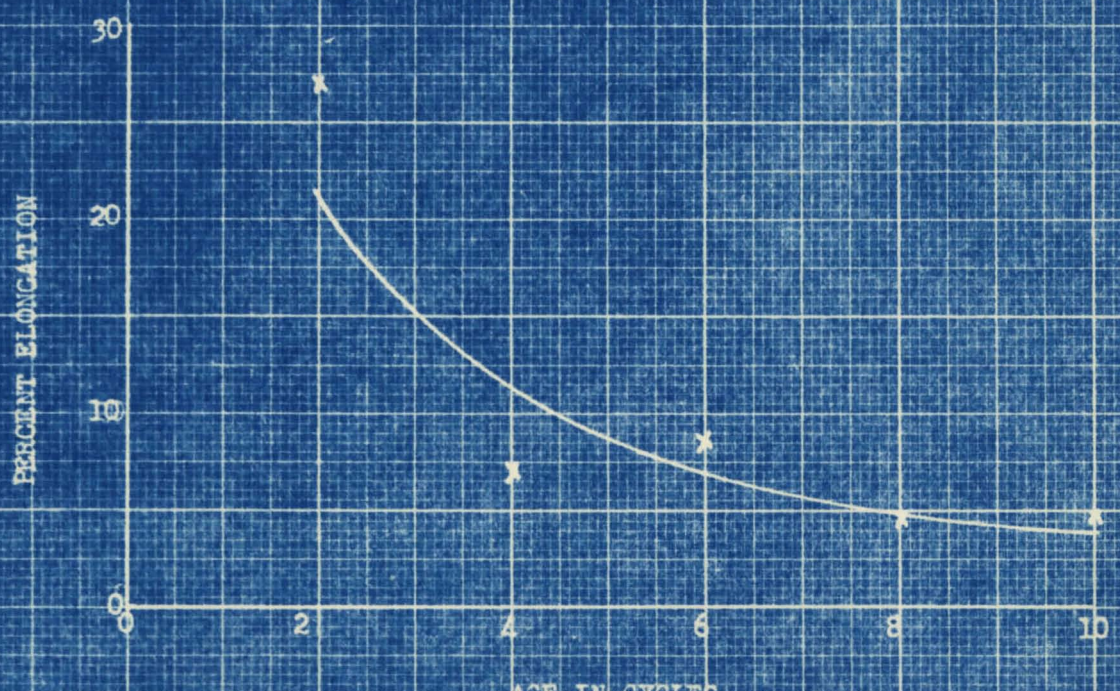

FIG. 7. PERCENT ELONGATION VS. AGE IN CYCLES OF ENAMEL AT 61 PERCENT REFLECTANCE

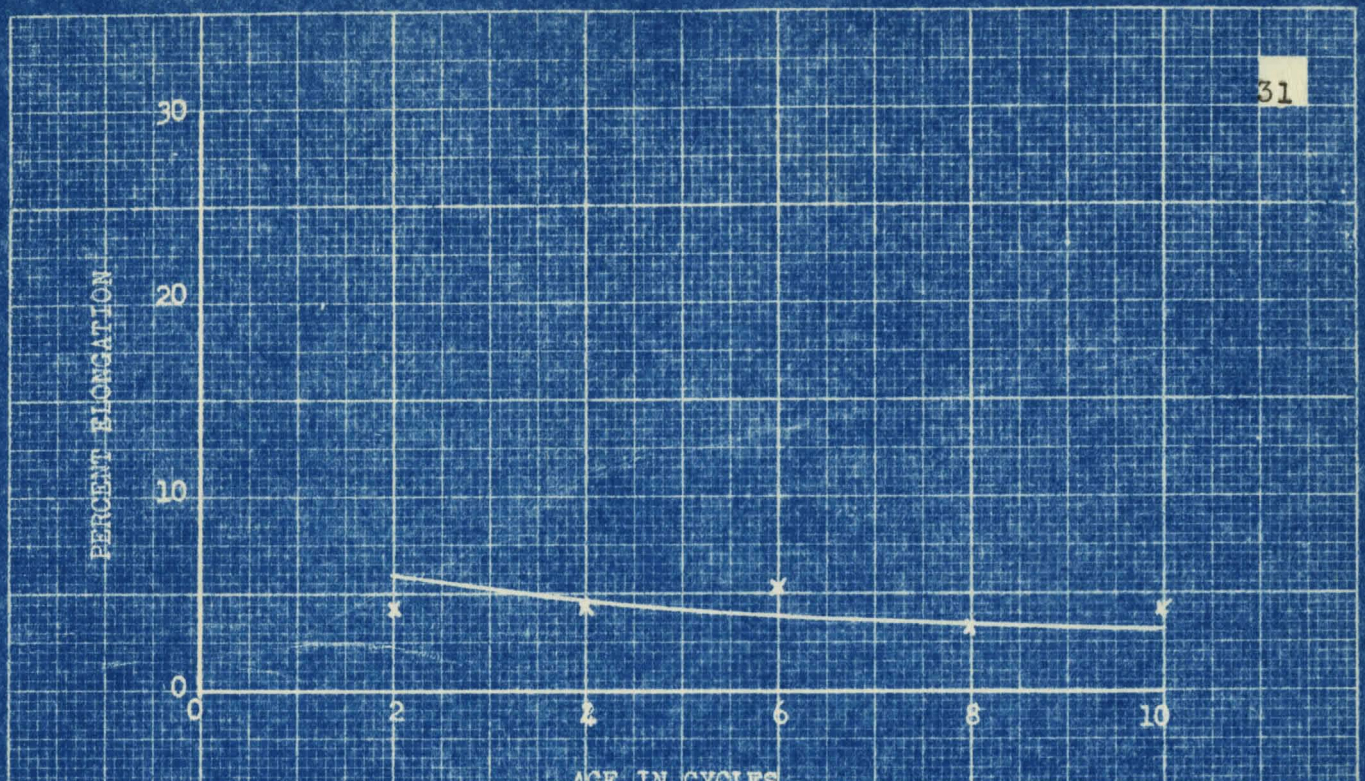

FIG. 8. PERCENT ELONGATION VS. AGE IN CYCLES OF ENAMEL AT 55 PERCENT REFLECTANCE

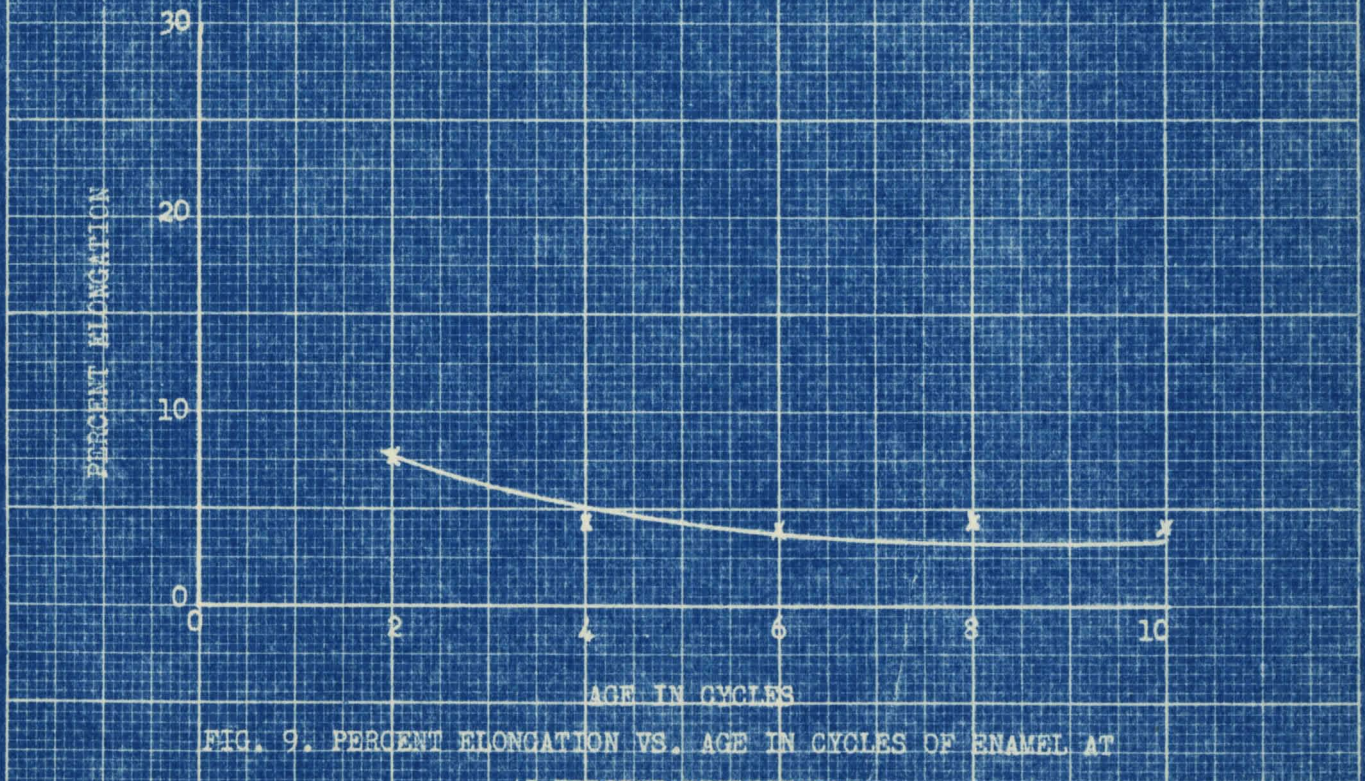

FIG. 9. PERCENT ELONGATION VS. AGE IN CYCLES OF ENAMEL AT 43 PERCENT REFLECTANCE

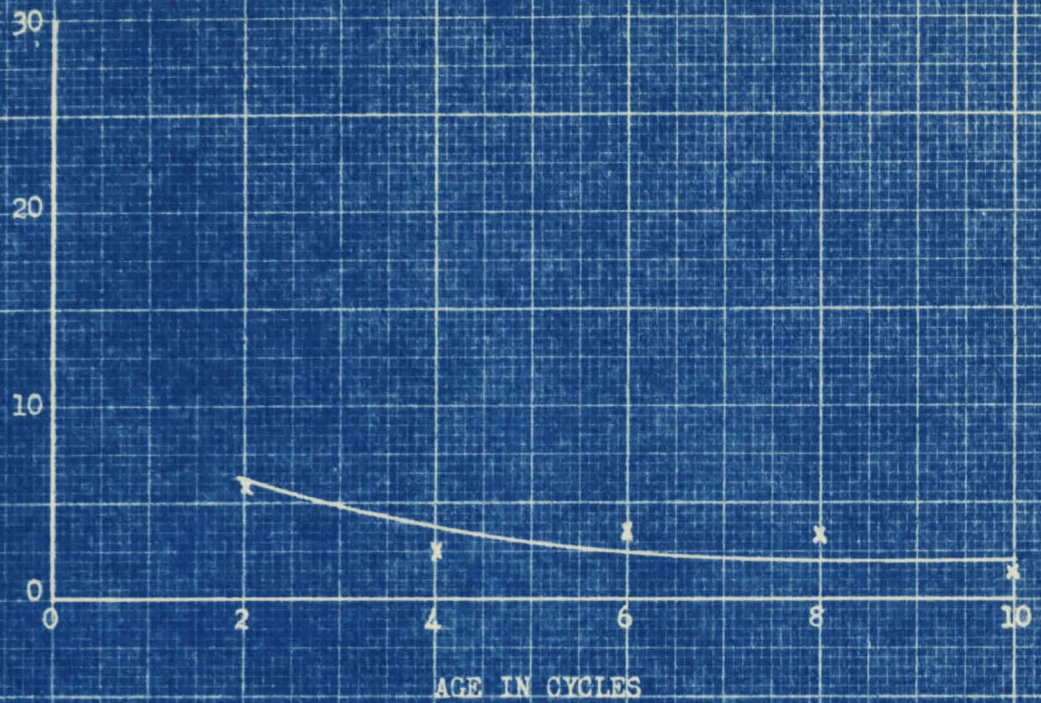

FIG. 10. PERCENT ELONGATION VS. AGE IN CYCLES OF ENAMEL AT 27 PERCENT REFLECTANCE

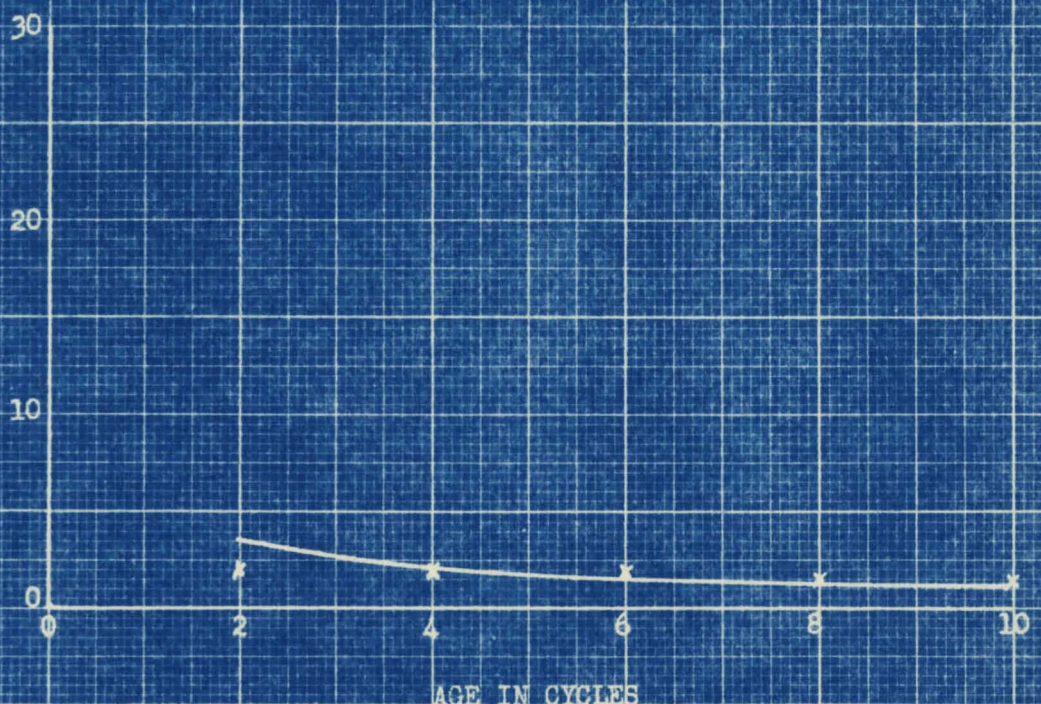

FIG. 11. PERCENT ELONGATION VS. AGE IN CYCLES OF ENAMEL AT 14 PERCENT REFLECTANCE

The other results were calculated from the experimental measurements according to the following methods. The elongation was read directly in percent from the testing machine indicator since the film was 100mm. long and the elongation was given on the indicator in mm. The volume percent unextractable was calculated by use of the formula developed by Ernst and Eudaley (5)

$$\text{Volume \% unextractables} = \left(1 - \frac{(Wex)(Wo+Wr+Wp)}{(Wf)(Vo+Vr+Vp)}\right)(100)$$

where Wex = weight of unextractable material, experimentally determined
Wf = weight of film sample
Wo = weight of oil in enamel sample
Wr = weight of resin in enamel sample
Wp = weight of pigment in enamel sample
Vo = volume of oil in enamel sample
Vr = volume of resin in enamel sample
Vp = volume of pigment in enamel sample

On examination of Figures 2-6, where percent reflectance is plotted against percent elongation at different ages it is shown that the percent elongation tends to increase as the percent reflectance increases. This would indicate that the more infrared absorbed the greater the rigidity and consequently the lower the distensibility.

Figures 7-11 show that at approximately constant reflection the percent elongation decreases rapidly only in one case and that at the higher reflectance. The values at 81% reflectance were not included since they were determined on a different testing machine and are not comparable.

Figures 12-17 give the customary type obtained

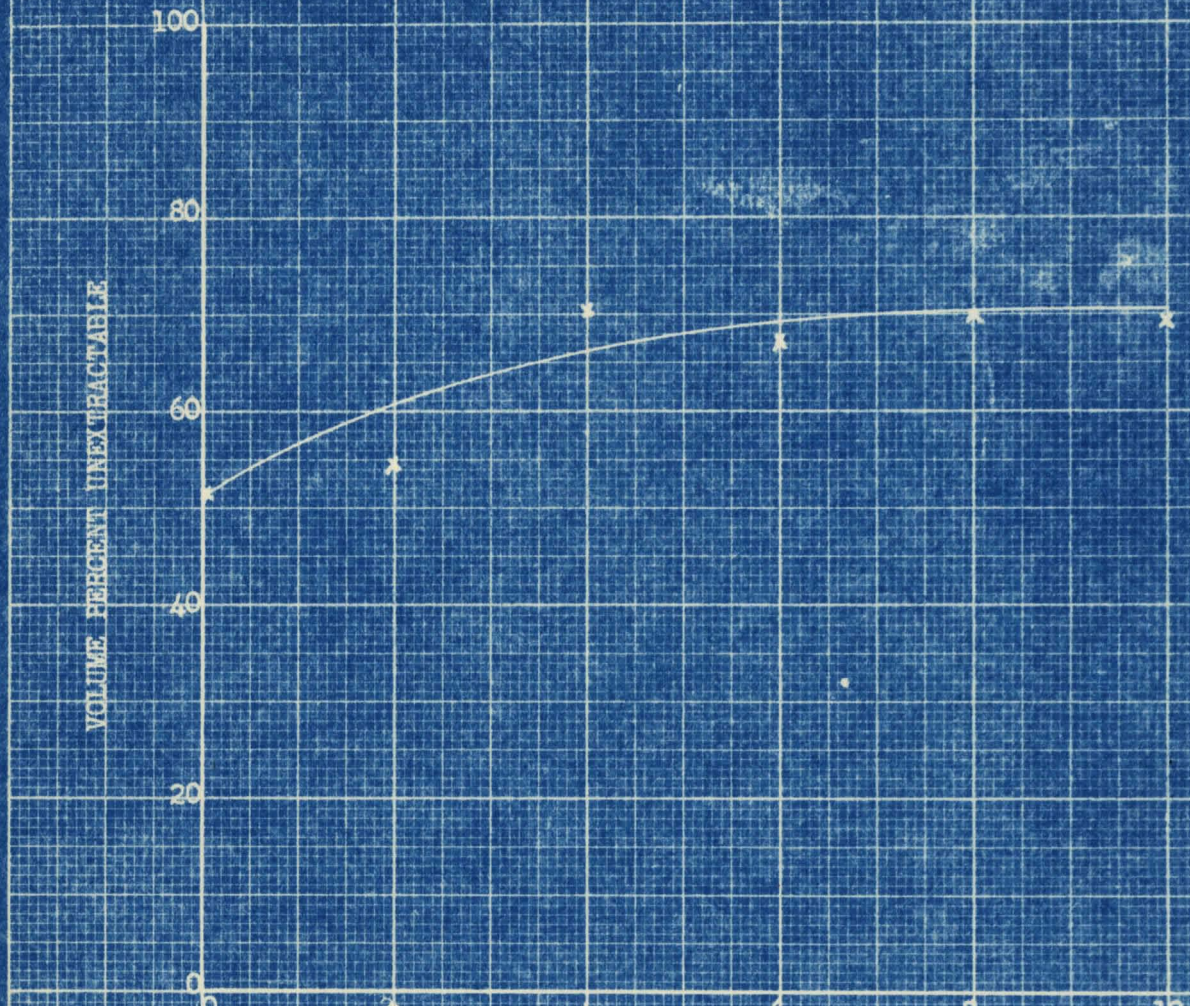

FIG. 12. VOLUME PERCENT UNEXTRACTABLE VS. AGE IN CYCLES OF ENAMEL AT 80 PERCENT REFLECTANCE

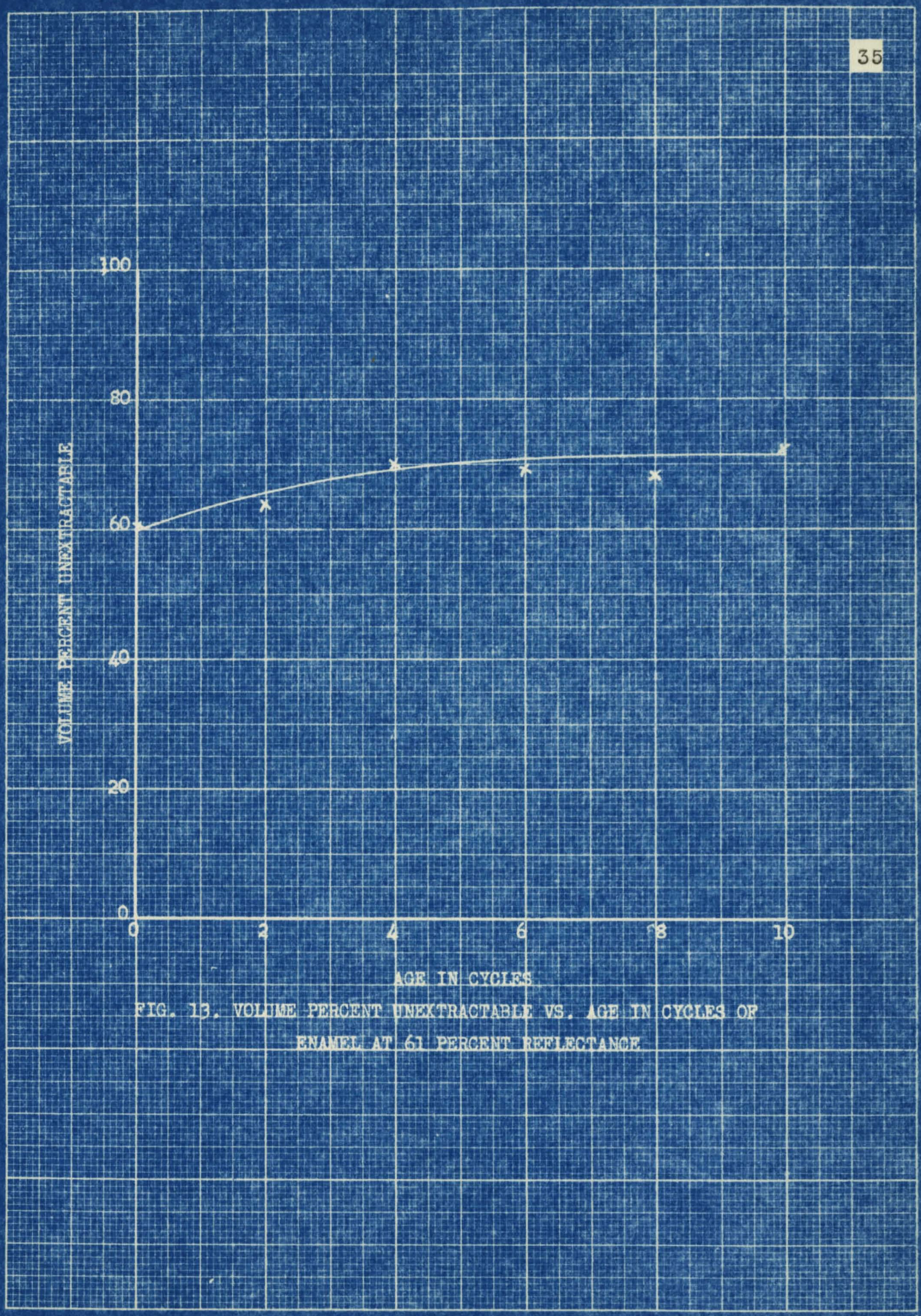

FIG. 13. VOLUME PERCENT UNEXTRACTABLE VS. AGE IN CYCLES OF ENAMEL AT 61 PERCENT REELECTANCE

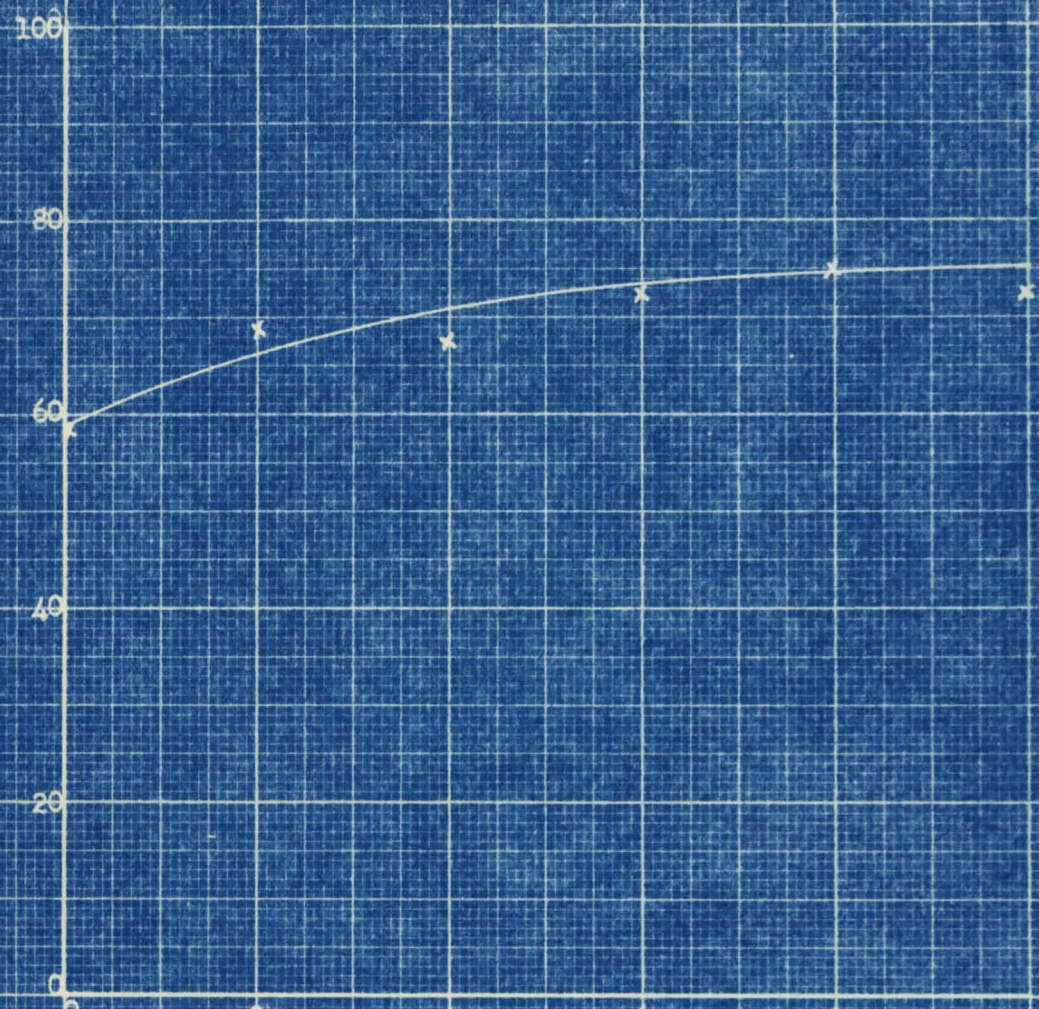

FIG. 14. VOLUME PRECENT UNEXTRACTABLE VS. AGE IN CYCLES OF ENAMEL AT 55 PERCENT REFLECTANCE

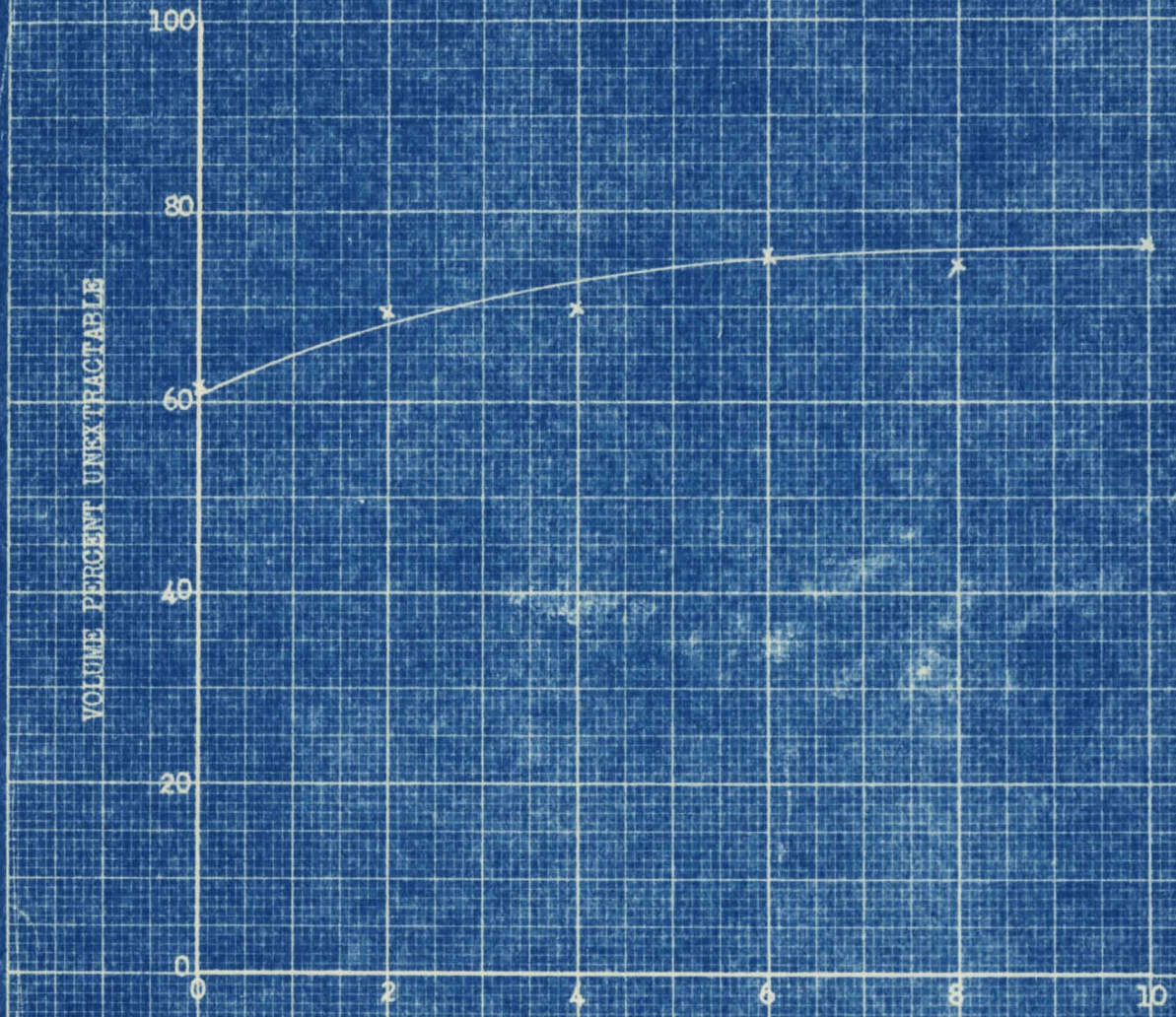

FIG. 15. VOLUME PERCENT UNEXTRACTABLE VS. AGE IN CYCLES OF ENAMEL AT 43 PERCENT REFLECTANCE

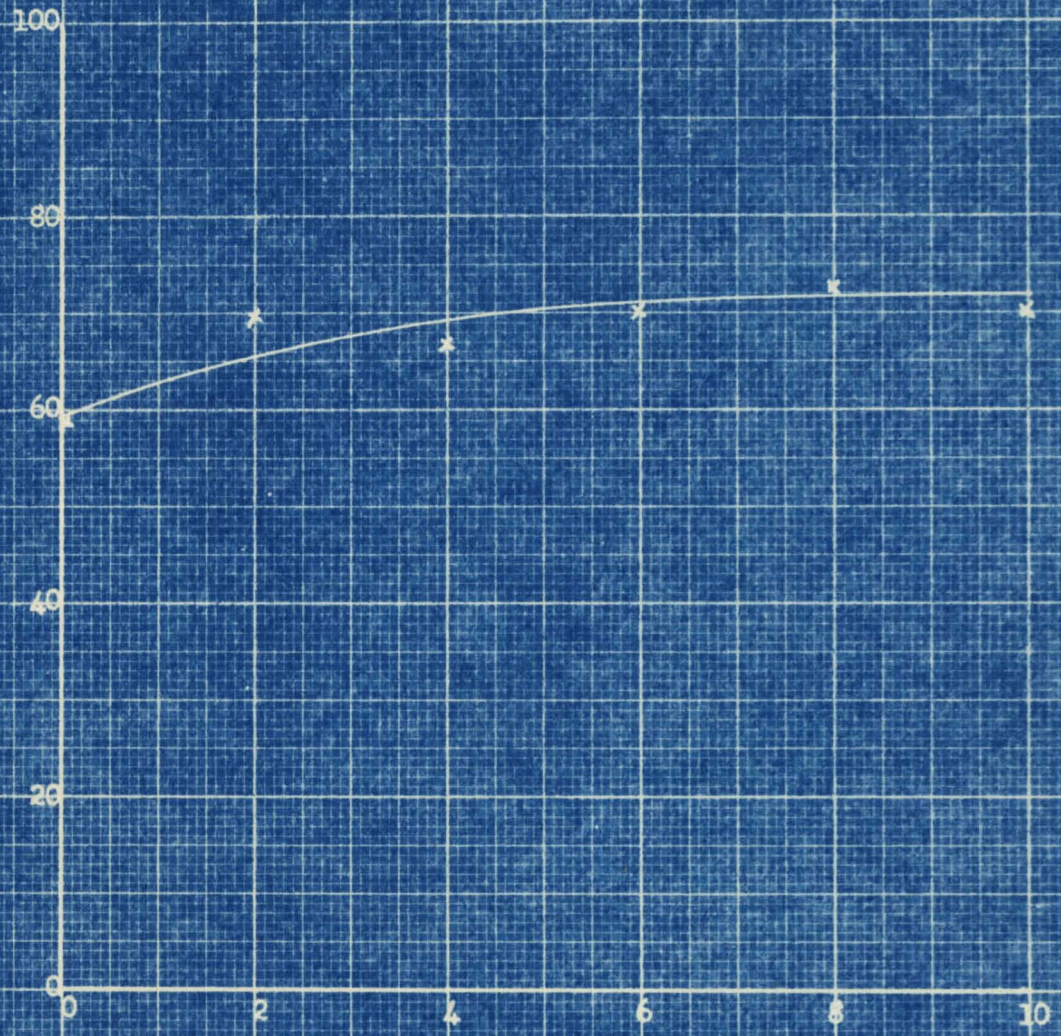

FIG. 16. VOLUME PERCENT UNEXTRACTABLE VS. AGE IN CYCLES OF ENAMEL AT 27 PERCENT REFLECTANCE

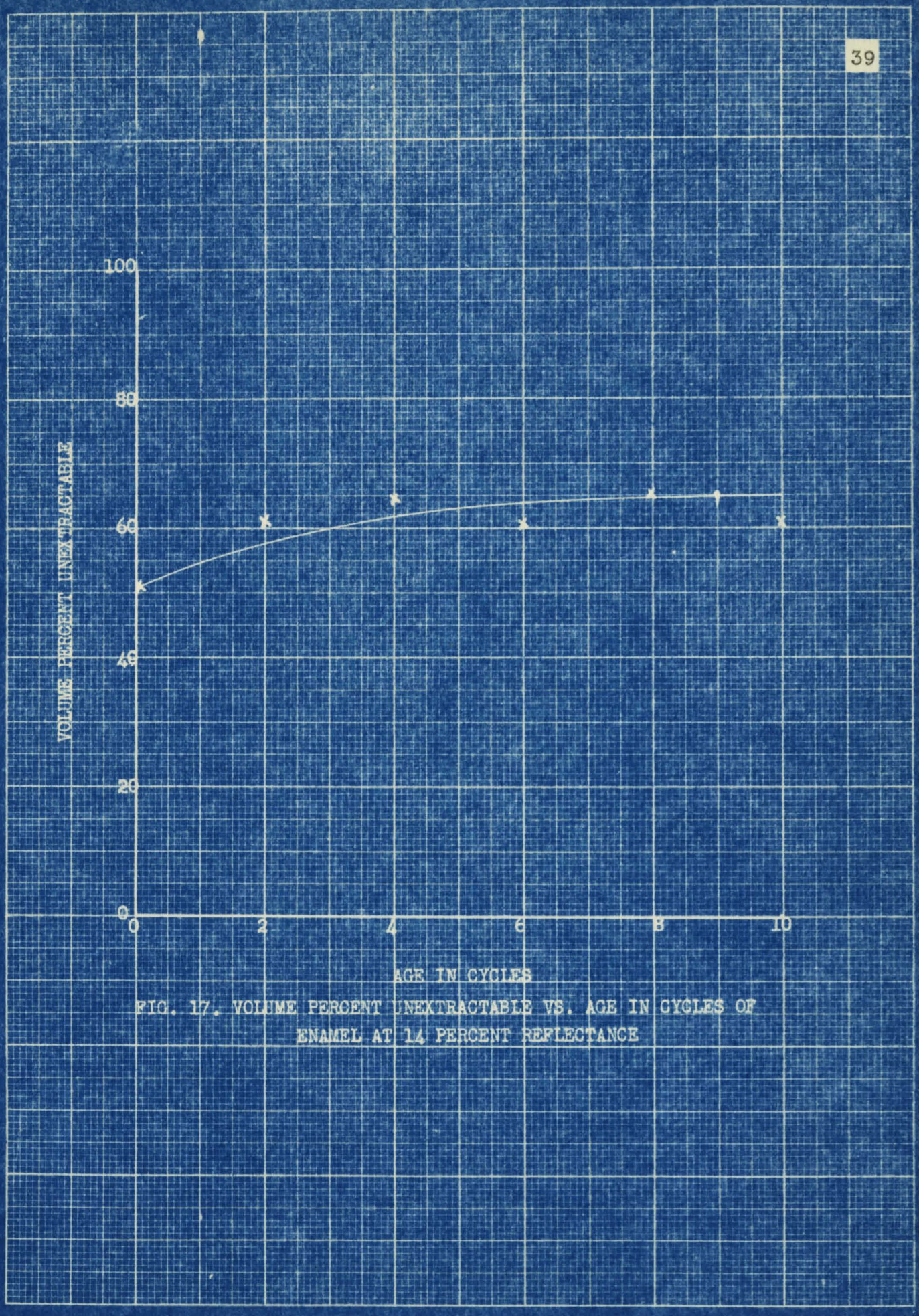

FIG. 17. VOLUME PERCENT UNEXTRACTABLE VS. AGE IN CYCLES OF ENAMEL AT 14 PERCENT REFLECTANCE

in previous investigations, however these curves rise more rapidly than that determined by Ernst and Eudaley (5) and do not reach the same ultimate polymer content. The rate of polymer formation is greatest for the films showing least percentage reflectance.

In general when age vs. volume percent un-extractable is plotted as in Figures 18-23, a parabolic shaped curve is obtained. They indicate that the maximum polymer content is reached at a reflectance of between 40 and 60 percent at comparable age.

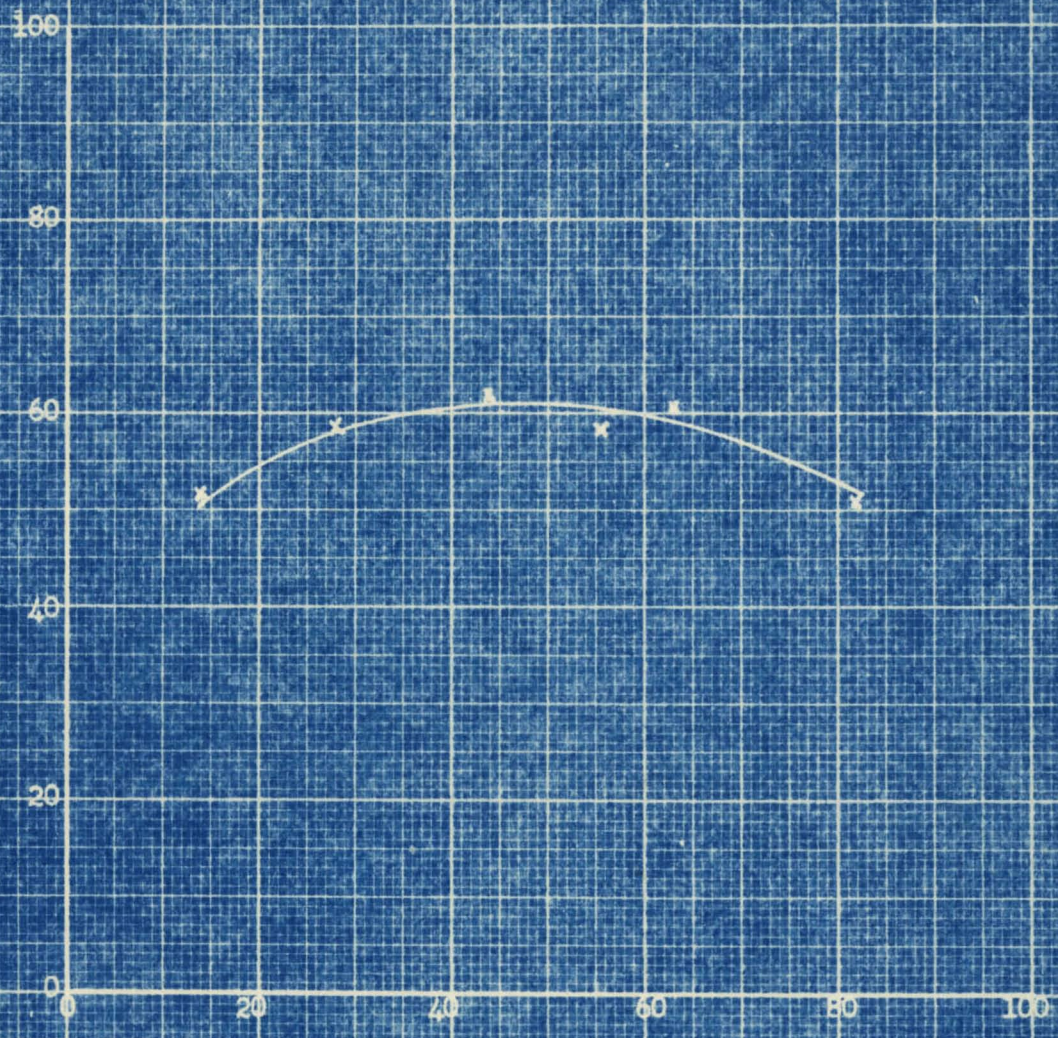

FIG. 18. VOLUME PERCENT UNEXTRACTABLE VS. PERCENT REFLECTANCE OF ENAMEL AT 0 CYCLES AGE.

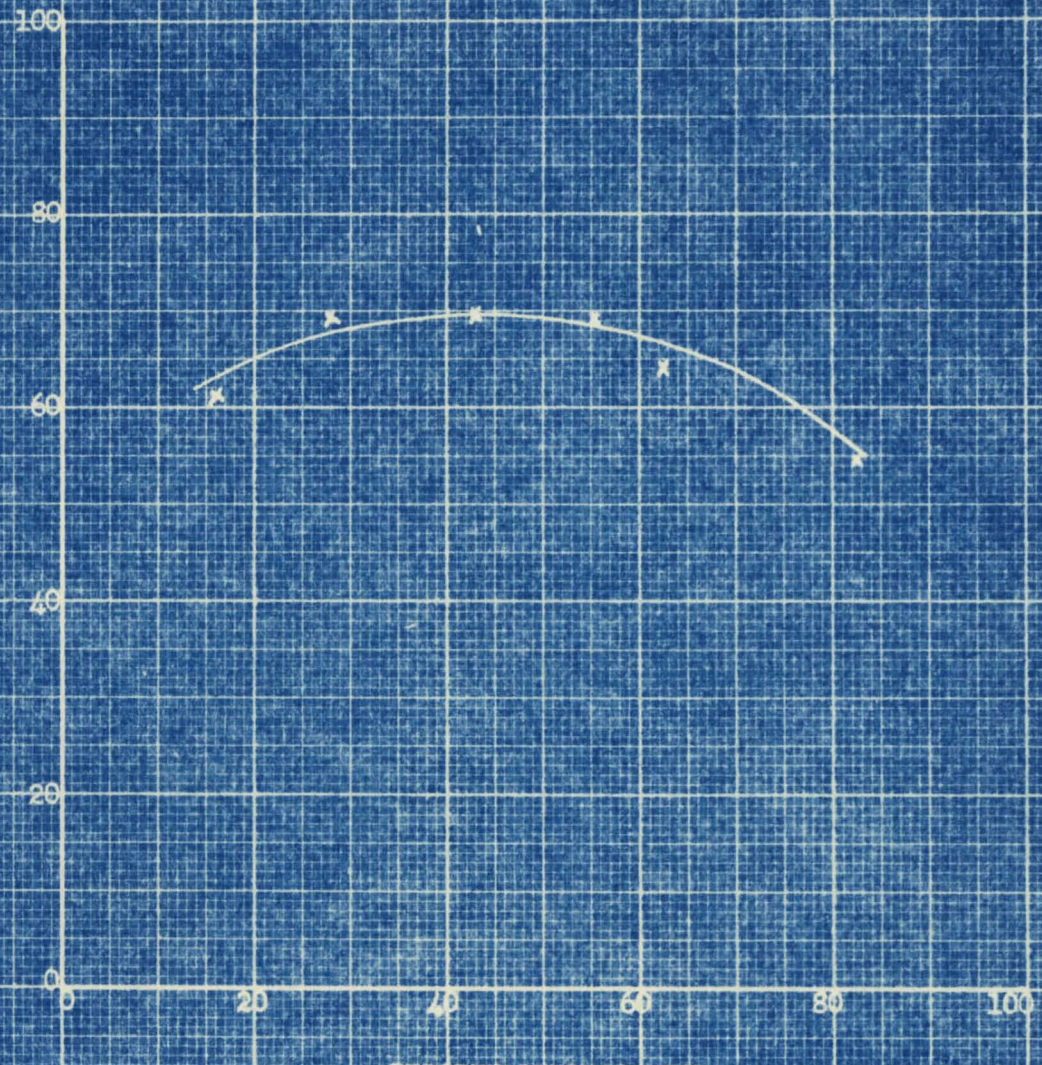

FIG. 19. VOLUME PERCENT UNEXTRACTABLE VS. PERCENT REFLECTANCE OF ENAMEL AT 2 CYCLES AGE

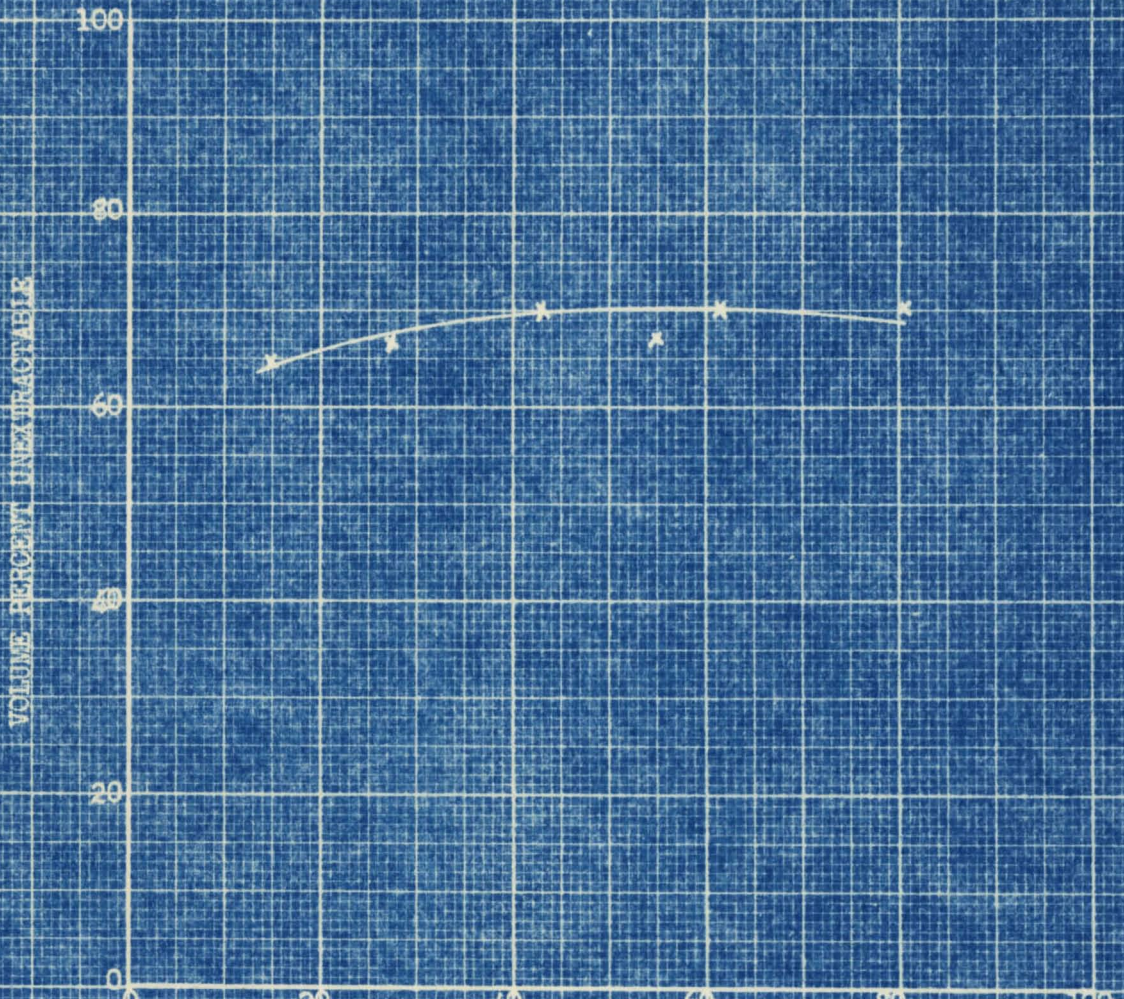

FIG. 20. VOLUME PERCENT UNEXTRACTABLE VS. PERCENT REFLECTANCE OF ENAMEL AT 4 CYCLES AGE

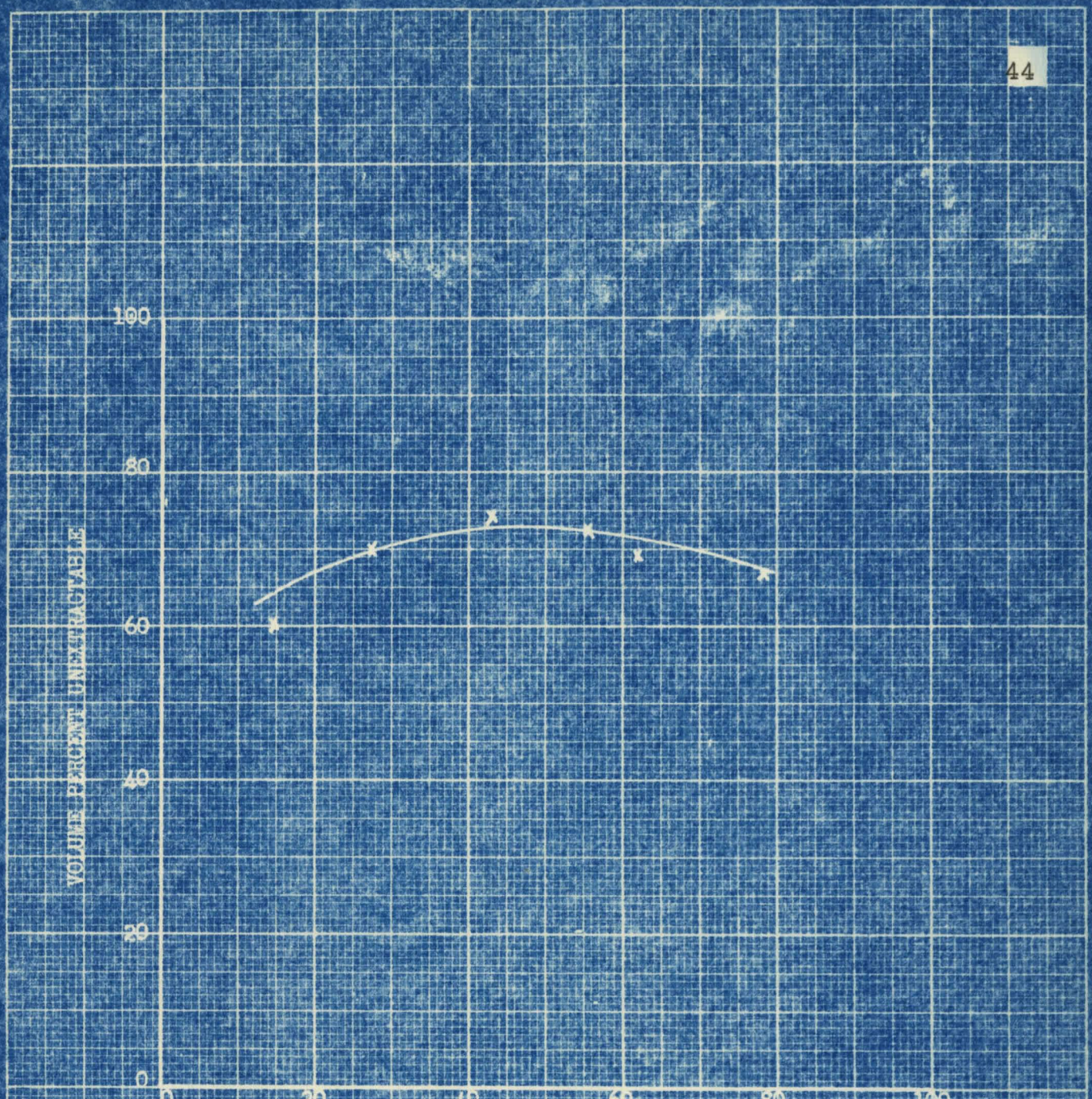

FIG. 21. VOLUME PERCENT UNEXTRACTABLE VS. PERCENT REFLECTANCE OF ENAMEL AT 6 CYCLES AGE

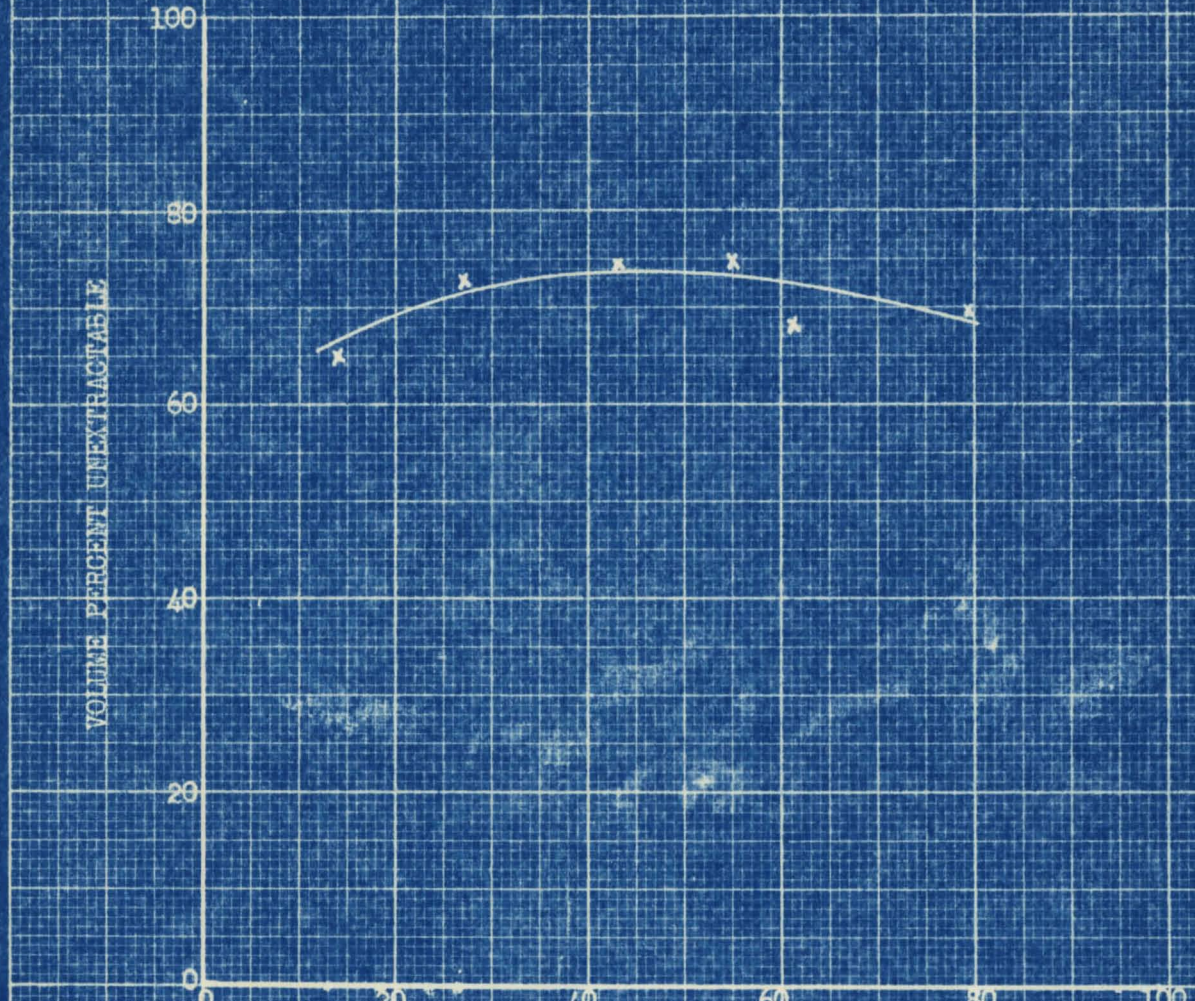

FIG. 22. VOLUME PERCENT UNEXTRACTABLE VS. PERCENT REFLECTANCE OF ENAMEL AT 8 CYCLES AGE

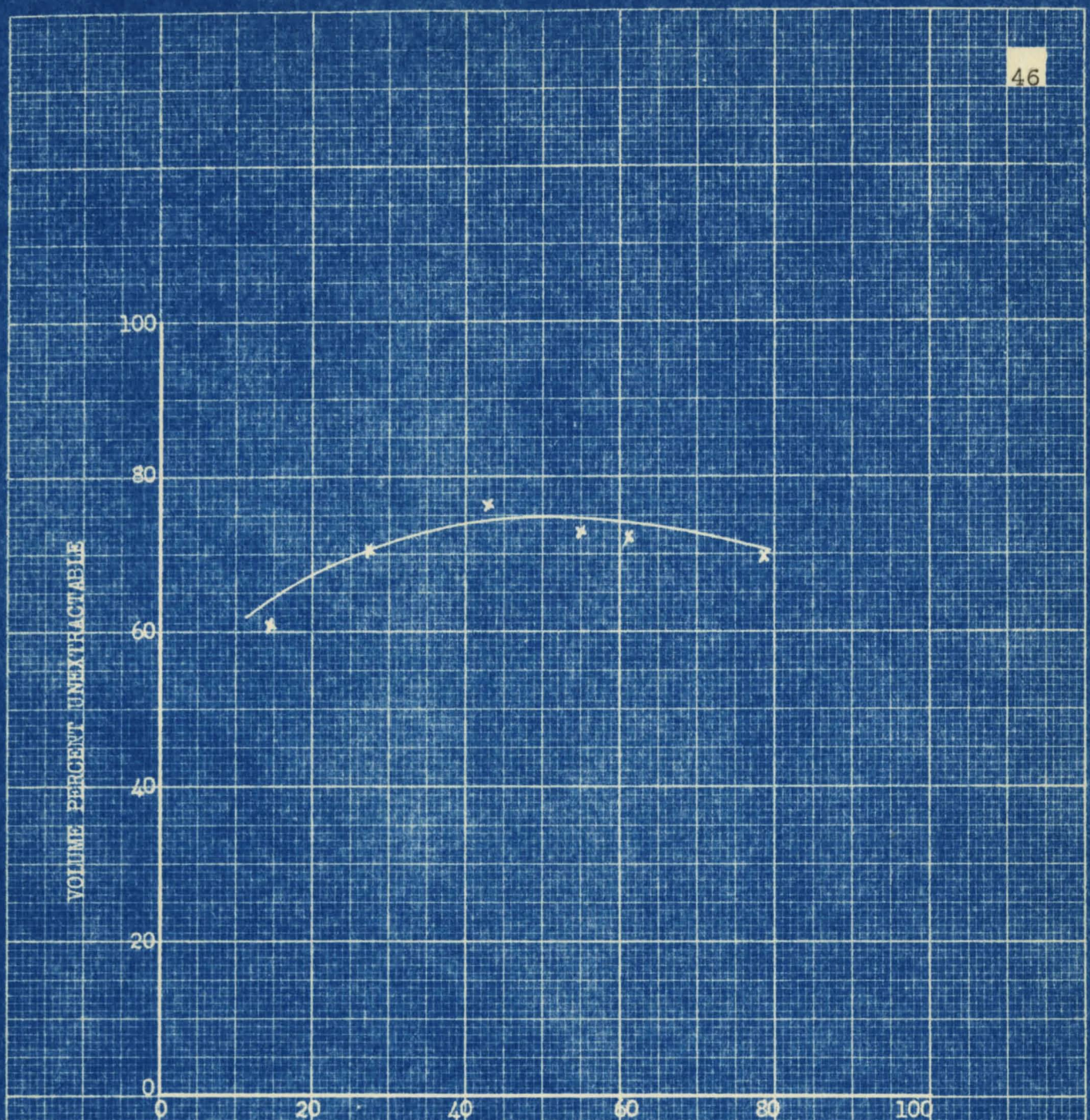

FIG. 23. VOLUME PERCENT UNEXTRACTABLE VS. PERCENT REFLECTANCE OF ENAMEL AT 10 CYCLES AGE

CONCLUSIONS

In correlating these results with the previous investigations and with information taken from the literature of other investigators, the following conclusions are reached.

(1) Distensibility of the film at various reflectances tends to approach a constant value as the life of the film increases, with the greatest distensibility being found in films of the highest reflectance. After the film has aged materially the variation in percent elongation is small over a large reflectance range but may be quite large in the early life of the film.

(2) Polymer content at constant reflectance changes materially during the age of the film, thus correlating well the distensibility data. The rate of polymer formation in the early life of a film is found to be greatest in those of least reflectance but does not reach so high an ultimate value as for the films of greater percentage reflectance. This suggests a breaking down of the original polymer formed at the lower reflectance as age increase to offset any further increase in polymer content.

(3) From an analysis of Figures 18 to 23 it appears that at comparable ages the polymer content of films with low reflectance is small either because of the antioxidant properties of the small amount of lampblack or because of a rupture of molecules due to the large amount of infrared emission absorbed or to a combination of both. As the percentage relfectance increases, the polymer content rises through a maximum which may again be attributed to a combination of the aforementioned effects. As the percentage reflectance increases there is a decrease in polymer content which may be caused by the failure of the film to receive enough radiation of a desirable type to activate the formation of additional polymers which would be formed by a certain quantity of infrared radiation.

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Brown Williamson Tobacco Co. Louisville

COMPREGNATION OF WOOD

TESTING OF DIMETHYLOLUREA PLASTIC

LIST OF TABLES

LIST OF FIGURES

ACKNOWLEDGMENT

The Author wishes to acknowledge the kind assistance and helpful guidance of Doctor G. C. Williams who directed the following research.

ABSTRACT

Four different woods are impregnated with a phenol-formaldehyde resin in aqueous solution. The density of the samples which had been cured at 100 and 1000 lbs. per sq. inch was determined at various positions.

It was found that a material increase of density was obtained in all woods investigated. No generalization can be made for change in density with or across grain in the woods examined. Dimensional stability is greatly increased by compregnation.

The combination of compression and impregnation to form compregnated woods offers a new approach to the production of high density woods.

INTRODUCTION

The purpose of this investigation is to determine the effect of impregnating wood with resins and subsequently curing the wood under pressure and at elevated temperature. An aqueous solution of resin is used and permitted to permeate the wood upon release of pressure while the wood is immersed in the resin solution. The effect of time of soak and the pressure under which the resin impregnated wood is cured is studied in relation to the final density.

HISTORICAL

Various methods of changing the density of wood by compression or impregnation have been tried. The latest method is the use of a combination of impregnation and compression, or as usually called 'compregnation'. In addition to raising the density of the wood it is also desirable to minimize any shrinking or swelling that might occur after the wood is in the final state.

Most investigations have been concerned with the use of resins to minimize the shrinking and swelling of thin layers of wood which were used to make plywood. In this field, the resin is used as an adhesive as well as an impregnating material.

Stamm and Seborg (2, 3, 4, 5) discuss the effect of various resin on the volume changes of wood and present results obtained when using resin treated wood in forming plywood, or laminated compressed wood. Super-pressed plywood has been investigated (1) by Bernhard, Perry, and Stern, this is a high density wood, a plywood in the true sense of the word, and derives its increase in density from the compression of the wood and the use of resin adhesives.

THEORETICAL

There are various methods of obtaining high density wood. One method resorts to the use of plywood and adhesives and processes under a high pressure; another method involving plywood is to impregnate the veneers with a resinous material and with high pressure and elevated temperature causes the resin to act as a bonding agent and adhesive. The bonding serves to prevent the pressed wood from exhibiting its elastic properties upon release of the pressure.

The method of compregnation, wherein the wood is first impregnated with a resinous material and then compressed, offers a new approach to the production of high density wood which will show small volume change when subjected to wide variations of moisture content of the surrounding atmosphere. Stamm and Seborg (3) report a definite minimizing of shrinking and swelling of wood treated with phenol-formaldehyde resins. The method of compregnation is to impregnate the wood with as large an amount as possible of a heat setting resin and subsequently to set the resin under pressure at elevated temperatures.

The method of treating wood with heat setting resins is to allow the resin to diffuse throughout the fibers of the wood and then by a heat treatment to convert them to the insoluble state. This serves the purpose of

making the wood less permeable to water and raises the density. The setting of the resin when the wood is compressed also serves to bind the fibers more firmly and therefore makes the wood less likely to undergo dimensional changes when pressure is released.

The methods of impregnating the wood with the resin solution may vary considerably. As in treating wood with preservatives, the wood may be subject to vacuum and then the resin solution forced into the wood under pressure and at elevated temperatures. Pressure alone may also be used to force the resin into the wood. Another method, which will be employed in this investigation, consists of saturating the wood with water, by continual soaking or employing slight pressure, the saturated wood is then subjected to high mechanical pressure (1000 psi.) to force the water out after which the pressure is released from the wood while it is immersed in a resin solution. Obviously this method depends upon the amount of water which can be replaced by the resin and the elastic properties of the wood since the magnitude of pressure differential created from the center to the outside of the wood depends upon how much the wood expands on release of pressure.

The amount of absorbed resin depends to a great extent upon the structure of the wood employed. Other

factors affecting the amount of solution absorbed is length of immersion, type of solution, viscosity of solution, and temperature. MacLean (6) discusses the various types of woods and gives a chart classifying the heartwood of various species with respect to penetrability. He points out that penetration may take place in three directions, namely, longitudinally, which is in the direction of the length of the tree trunk; radially, which is in the direction of the radius of the tree; and tangentially or circumferentially, which is in the direction of the annual rings. Generally the greatest penetration is found to be longitudinally because of the smaller number of cell walls necessary to penetrate. From data of MacLean the magnitude of penetration should decrease in the following order, red oak, cottonwood, and white oak.

Erickson, Schmitz, and Gortner (7) have obtained data on the permeability of various woods and they show that white oak heartwood is one of the least permeable woods. They used pressure differentials up to 20 lbs. per sq. inch across a piece of wood one cm. thick. The amount of solution absorbed may be increased by partial rupture of cell wall when subjected to high pressure thus decreasing materially the resistance to penetration.

EXPERIMENTAL

APPARATUS

The main piece of apparatus used was a hydraulic press with thermostatically operated, electrically heated platens. This press, made by the Charles E. Francis Company, is hand operated with platens one foot square and a piston of 50 square inches surface area. It is possible to obtain up to 50 tons total load on this press. The press is supplied with a pressure gauge reading from 0 to 2000 lbs. per sq. inch on the surface of the piston and graduated in 40 lb. increments.

Other apparatus included a clamp for holding the pressure on the wood when it was transferred to the resin solution for the release of pressure. The lower part of the clamp is a piece of steel which has bolts placed in each corner, heads flush with the surface. The upper part of the clamp has a raised and machined surface on which pressure can be applied. Two slots in each end of the upper part were so placed that the bolts from the lower piece could fit into them. The sample was placed in the clamp and pressure applied, the pressure being retained on the wood by tightening the nuts on the bolts. A shallow pan, slightly larger than the clamp was used to hold the resin solution as the pressure was released from the wood. A Sargent Electric Drying Oven was used to pre-cure the samples.

A pressure cylinder was used to saturate the wood with water, a pressure of 15 lbs. per square inch used, and the samples soaked up to 72 hours.

RAW MATERIALS

Samples of cottonwood, willow, and red and white oak were used. They were approximately 4" by 3/7-16 " by 1" before soaking. The resin used for impregnation was Amberlite FR 23, a phenol- formaldehyde resin manufactured by Resinous Products and Chemical Company. This resin is water soluble and was used in aqueous solution in concentration of 30% solids content by weight.

PROCEDURE

The wood samples were placed in a pressure vessel, water of about 140° F added, and 15 lbs. air pressure applied. The water was changed frequently. The samples were weighed before and after soaking with water. The samples were pressed to one-half their original size, if possible, or until a maximum of 1000 lbs. per sq. inch was reached, they were then clamped in the holder, placed under the resin solution for a period of 1,5,10,30, and 60 minutes and 12 hours. The samples were weighed after air drying, pre-cured in an oven at 140° F for 10-12 hours and again weighed.

After oven drying the samples were placed in the hot press, at a temperature of 250° F and pressure applied , 100 and 1000 lbs. per sq. inch were used. The samples were held at this temperature for 20 minutes and then allowed to cool in the press to 175° F.

The compregnated samples were then sawn to produce 1/2 inch squares for determination of density. The samples for density determination were so chosen as to give three determinations with the grain and two across the grain, see Figure 1, in addition to a sample from the center of the block. Dimensions of the cubes were taken with a vernier caliper, and they were weighed on an analytical balance.

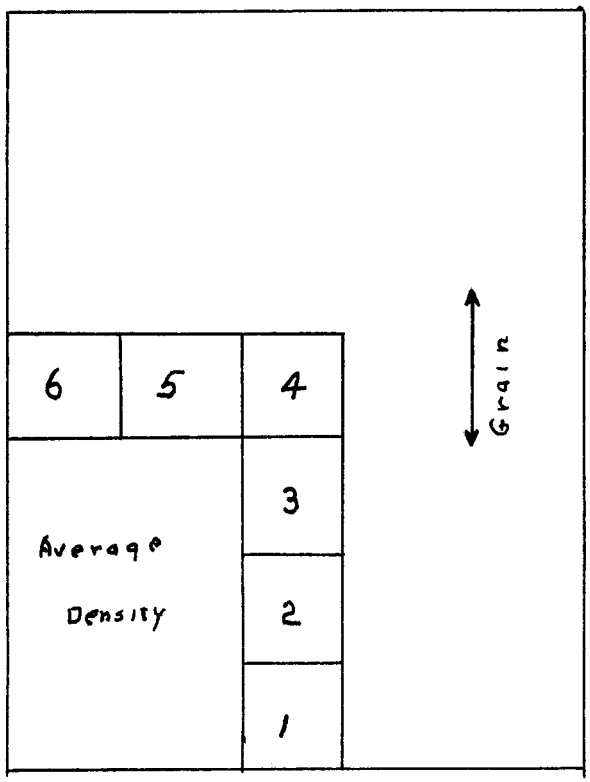


Fig. 1. Position of Samples taken for Density Determinations

DENSITY OF WOODS CURED AT 1000 LBS. PER SQ. INCH

WOOD	TIME OF SOAK MINUTES	AVERAGE DENSITY	DENSITY AT POSITION 1	2	3	4	5	6
COTTONWOOD	1	0.917	0.983	0.978	0.946	0.939	0.902	0.869
	5	0.979	1.068	0.981	0.959	0.983	0.939	0.948
	10	0.956	1.025	0.977	0.956	0.966	0.946	0.899
	30	1.015	1.075	0.976	0.872	0.981	0.989	0.956
	60	1.133	1.300	1.210	1.200	1.195	1.158	1.085
	720	1.162	1.280	1.168	1.125	1.112	1.132	1.062
RED OAK	1	1.235	1.178	1.235	1.292	1.289	1.340	11.368
	5	1.075	1.085	1.110	———	———	1.152	1.065
	10	1.005	1.035	1.009	1.090	1.080	1.070	0.998
	30	1.015	1.172	1.237	1.251	1.278	1.265	1.105
	60	1.133	1.133	1.180	1.258	1.305	1.223	1.151
	720	1.162	1.178	1.200	1.213	1.227	1.192	1.085
WILLOW	1	0.948	1.038	1.182	1.190	1.230	1.175	0.936
	5	1.003	1.044	1.033	1.072	1.067	1.065	1.978
	10	1.011	1.011	1.043	1.145	1.197	1.172	1.059
	30	1.137	1.118	1.215	1.190	1.187	1.153	1.145
	60	1.023	1.084	1.190	1.241	1.228	1.218	1.038
	720	1.190	1.192	1.228	1.208	1.205	1.200	1.102
WHITE OAK	1	1.142	1.115	1.160	1.160	1.185	1.188	1.135
	5	1.138	1.161	1.182	1.212	1.203	1.145	1.145
	10	1.175	1.162	1.178	1.193	1.185	1.310	1.175
	30	1.210	1.230	1.212	1.288	1.288	1.258	1.195
	60	1.200	1.230	1.282	1.245	1.258	1.152	1.188
	720	1.062	1.077	1.096	1.110	1.182	1.136	1.082

DENSITY OF WOODS CURED AT 100 LBS. PER SQ. INCH

WOOD	TIME OF SOAK MINUTES	AVERAGE DENSITY	DENSITY AT POSITION					
			1	2	3	4	5	6
COTTONWOOD	1	0.581	0.556	0.566	0.570	0.569	0.563	0.542
	5	0.582	0.573	0.570	0.630	0.574	0.561	0.572
	10	0.702	0.730	0.734	0.734	0.723	0.722	0.701
	30	0.770	0.738	0.760	0.873	0.736	0.744	0.703
	60	0.763	0.755	0.765	0.842	0.722	0.706	0.720
RED OAK	1	0.861	0.868	0.864	0.865	0.863	0.868	0.848
	5	0.805	0.806	0.812	0.810	0.799	0.806	0.805
	10	0.826	0.798	0.820	0.819	0.804	0.816	0.820
	30	0.783	0.805	0.804	0.809	0.811	0.794	0.799
	60	0.789	0.816	0.829	0.829	0.792	0.783	0.767
	720	0.938	0.953	0.952	0.959	0.939	0.949	0.931
WILLOW	1	0.692	0.673	0.659	0.712	0.668	0.674	0.685
	5	0.702	0.694	0.699	0.719	0.687	0.690	0.669
	10	0.810	0.803	0.863	0.811	0.793	0.796	0.770
	30	0.776	0.802	0.798	0.816	0.817	0.806	0.766
	60	0.723	0.719	0.728	0.787	0.719	0.714	0.702
	720	0.746	0.726	0.735	0.808	0.717	0.720	0.700
WHITE OAK	1	0.807	0.790	0.769	0.792	0.787	0.788	- - -
	5	0.801	0.788	0.801	0.799	0.791	0.803	0.790
	10	0.816	0.802	- - -	- - -	0.813	0.819	0.816
	30	0.832	0.831	0.836	0.839	0.836	0.836	0.827
	60	0.846	0.830	0.825	0.824	0.816	0.837	0.835
	720	0.830	0.903	0.909	0.919	0.907	0.904	0.870

RESULTS AND INTERPRETATIONS

From Figures 2 to 5, it is evident that within experimental error the density increases with the time of soaking. The change in density with time is slightly more rapid for the material treated at the higher pressure, this is probably not due entirely to the amount of resin absorbed but may be attributed in part to the materials of highest resin content being most easily pressed.

The increase in density is found to be greatest in the cottonwood, followed by willow, white and then red oak. These results were to be expected since the structure of the willow and cottonwood is such that a large amount of material may be taken in among their fibers. The red and white oak has a grain structure which offers great resistance to the permeation of liquids.

Upon inspection of Figures 6 to 9, it appears that no generalization can be made for the entire group. At 1000 lbs. per sq. inch pressure the willow alone gives a decrease in density as the center if approached along the grain, this is to be expected under normal circumstances. In the other samples, both with and across the grain at 1000 lbs. per sq. inch, the density is in general found to be greatest at the center and dropping off rapidly as the edge is approached. This may be attributed to several

FIG. 2 TIME OF SOAK VS. AVERAGE DENSITY

WILLOW

x - 1000 lbs. per sq. inch

o - 100 lbs. per sq. inch

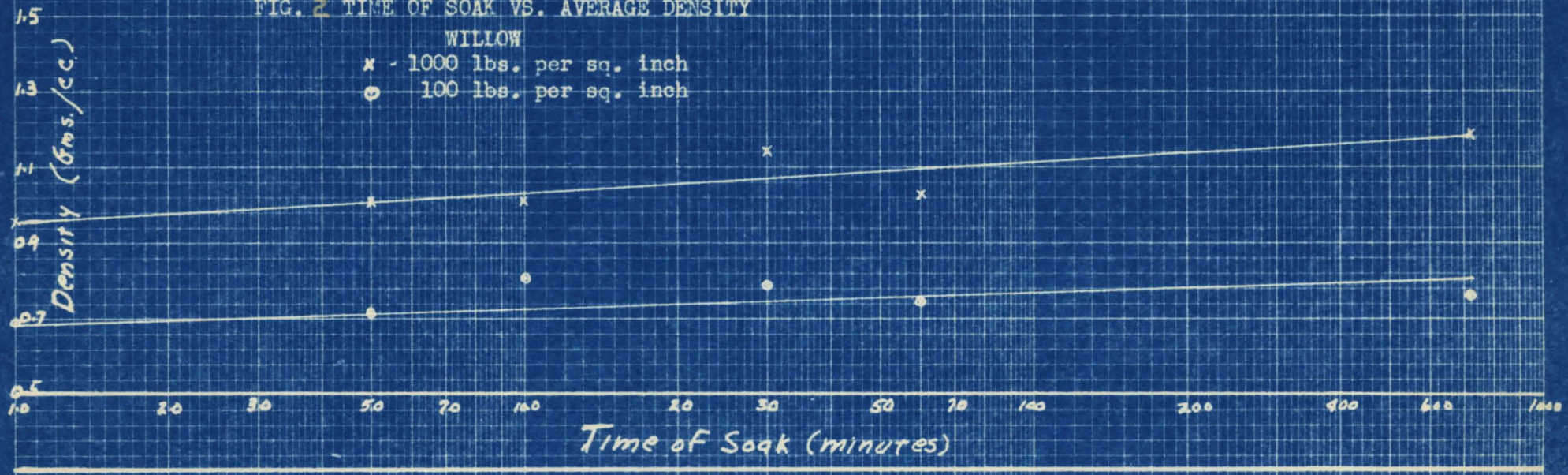


FIG. 3 TIME OF SOAK VS. AVERAGE DENSITY

WHITE OAK

x - 1000 lbs. per sq. inch

o - 100 lbs. per sq. inch

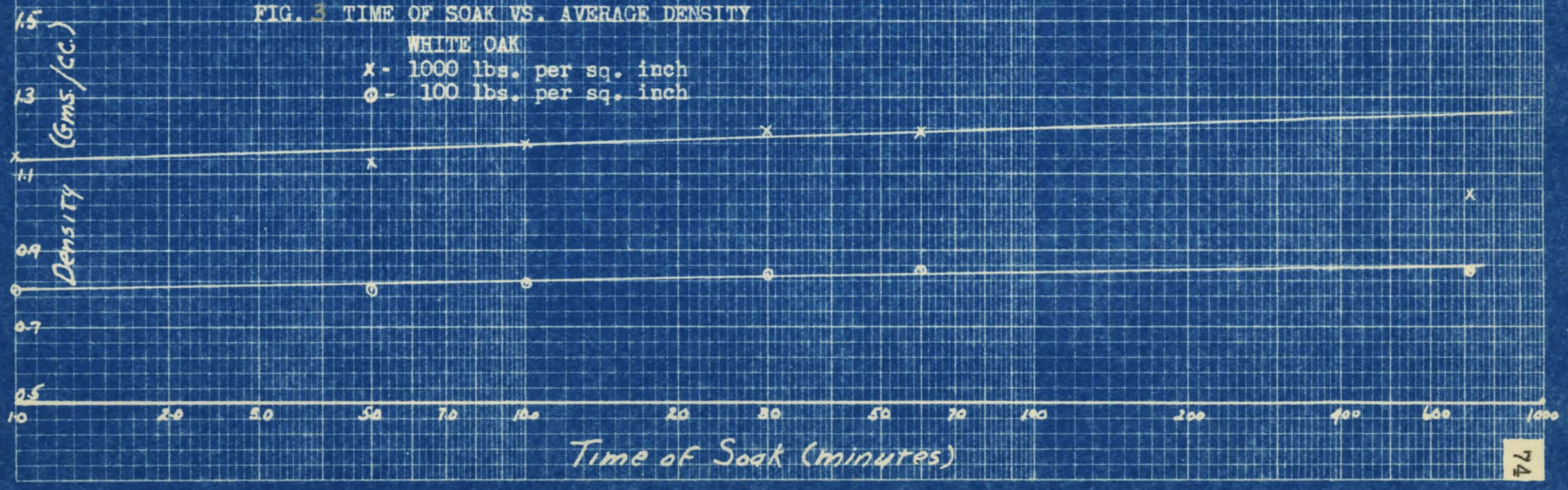


FIG. 4 TIME OF SOAK VS. AVERAGE DENSITY
COTTONWOOD
x - 1000 lbs. per sq. inch
o - 100 lbs. per sq. inch

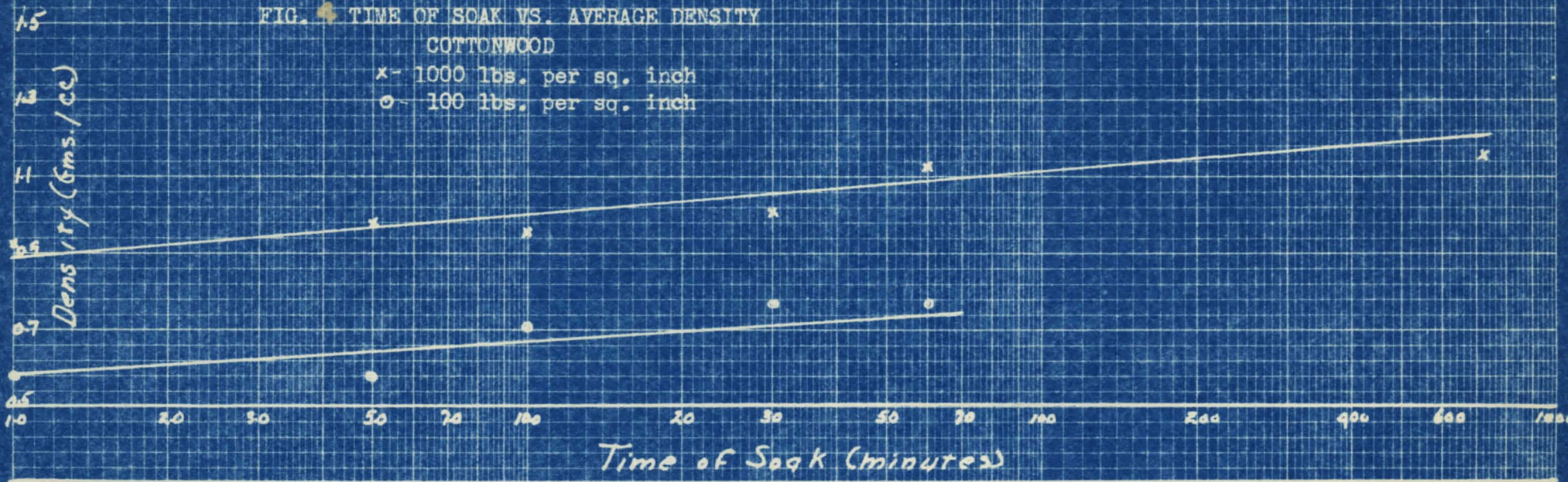
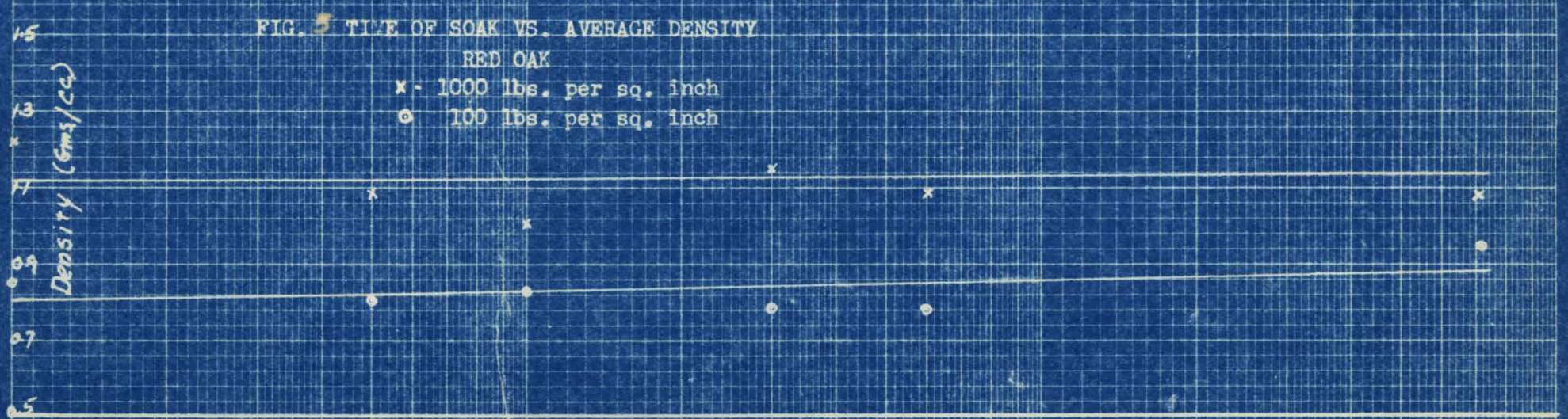
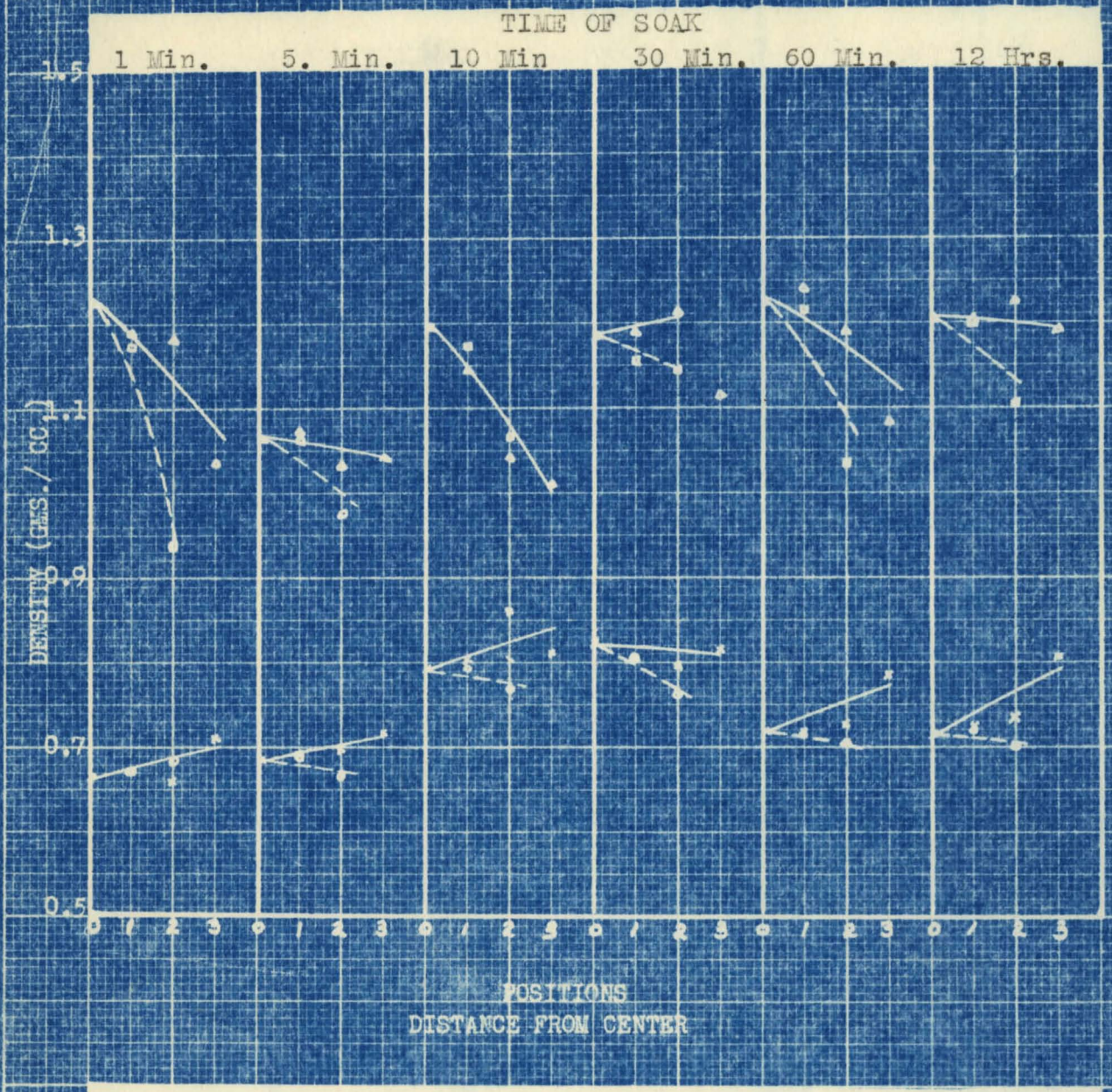


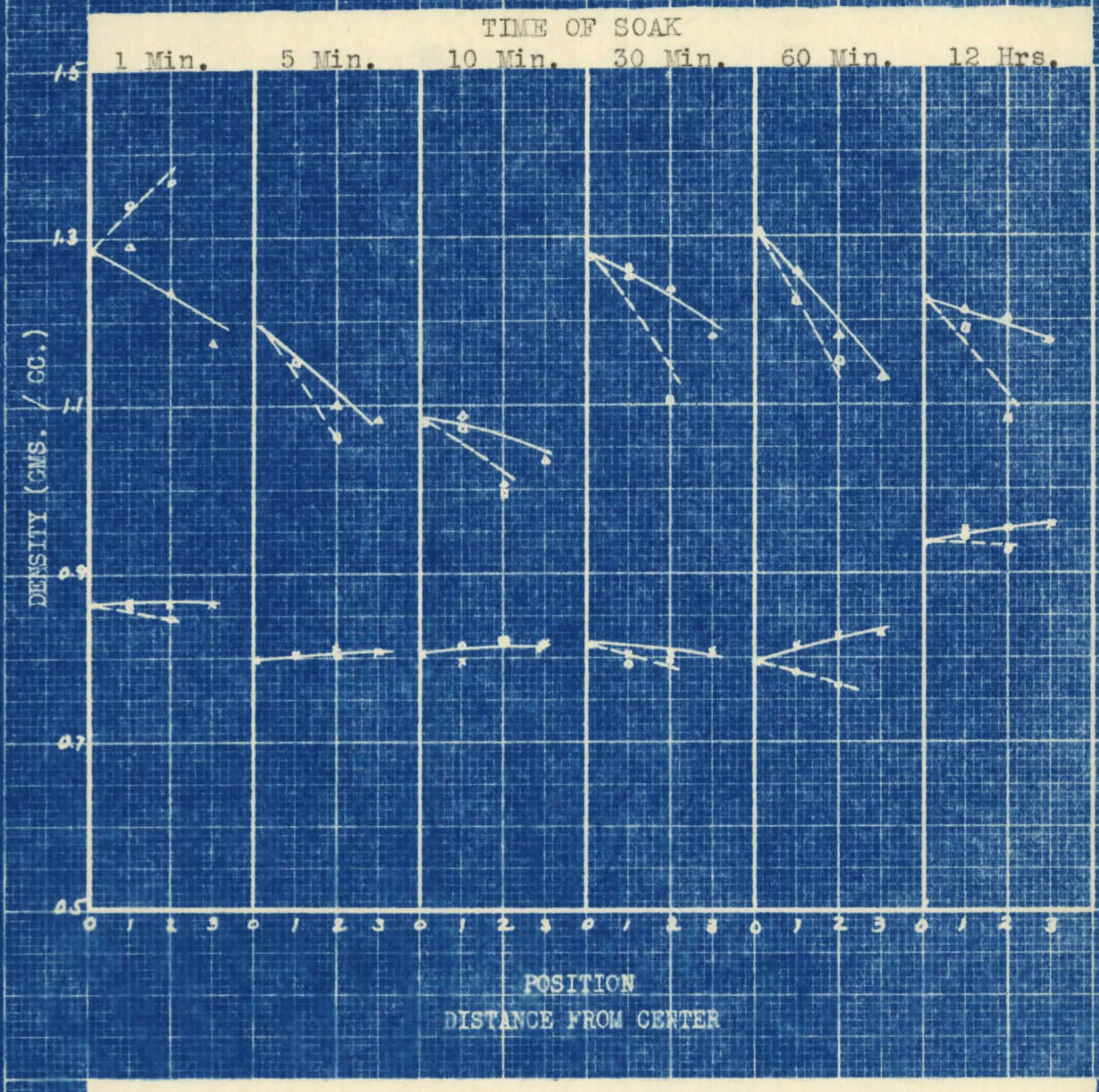
FIG. 5 TIME OF SOAK VS. AVERAGE DENSITY
RED OAK
x - 1000 lbs. per sq. inch
o - 100 lbs. per sq. inch





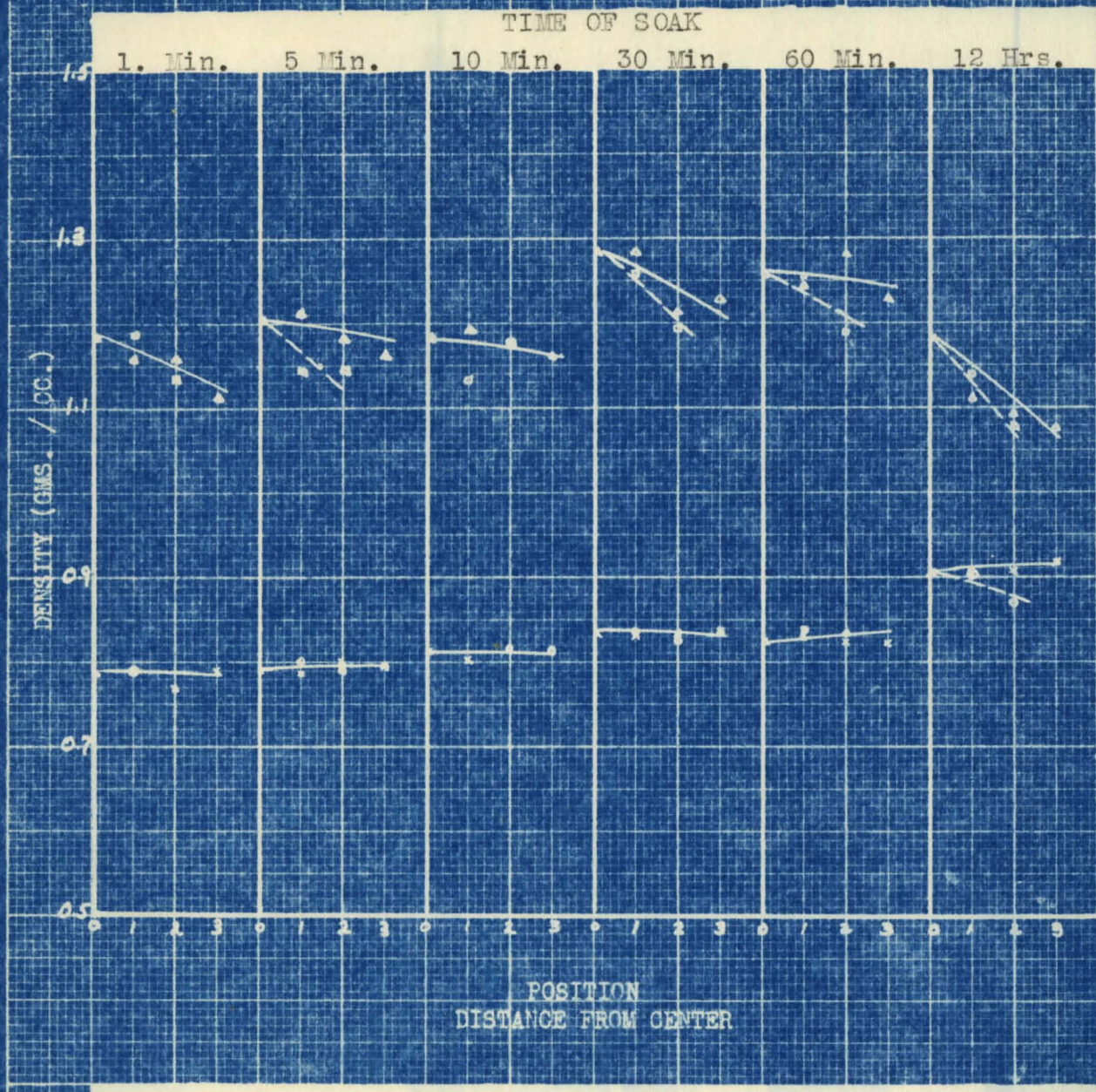
△ with grain, 1000 lbs. per sq. inch
□ across grain, 1000 lbs. per sq. inch
× with grain, 100 lbs. per sq. inch
○ across grain, 100 lbs per sq. inch

Fig. 6. Position vs. Density for Willow at Various lengths of soaking.



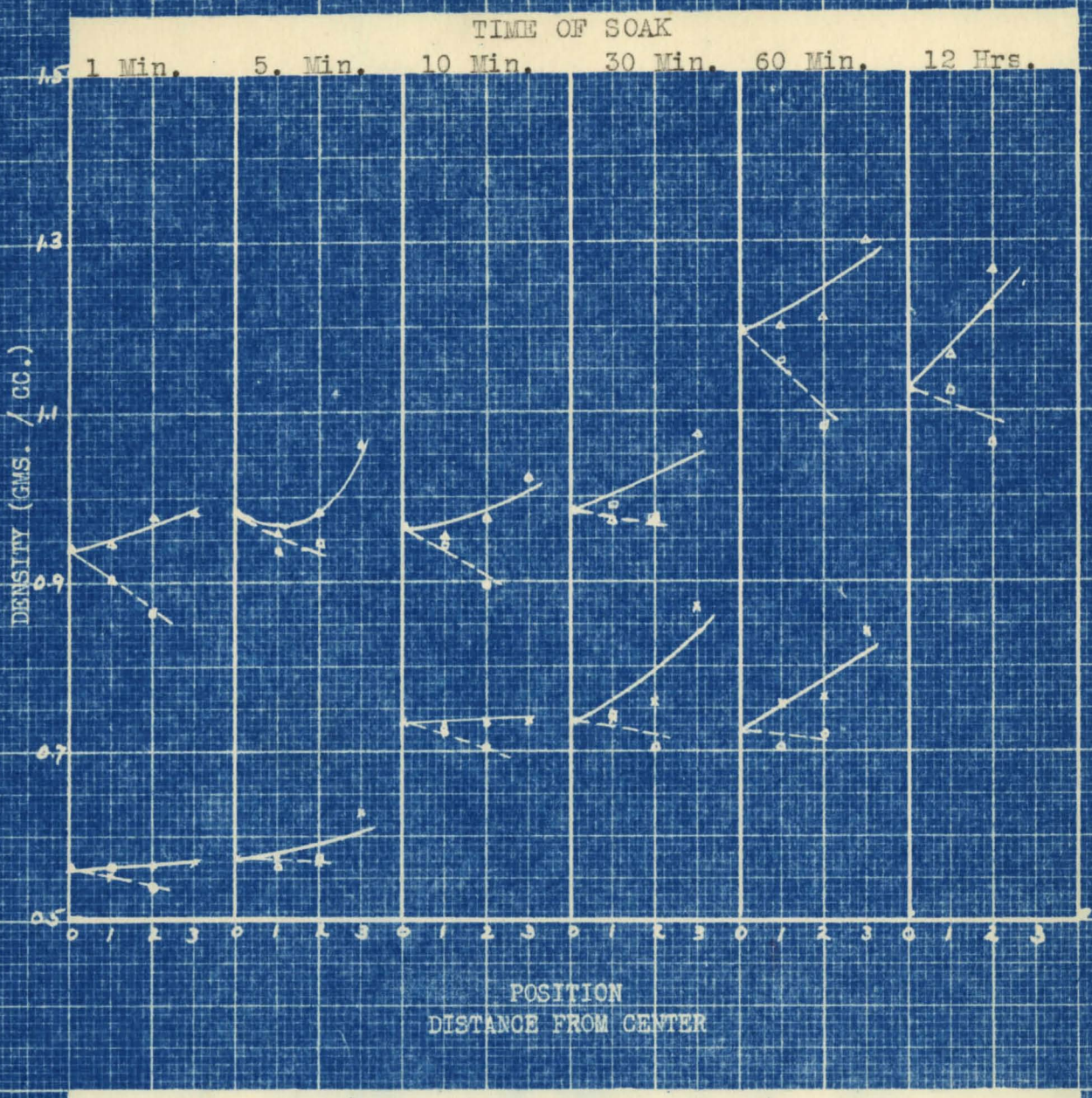
△ with grain, 1000 lbs. per sq. inch
□ across grain, 1000 lbs. per sq. inch
× with grain, 100 lbs. per sq. inch
○ across grain, 100 lbs. per sq. inch

Fig. 7. Position vs. Density for Red Oak at Various lengths of soaking.



△ with grain, 1000 lbs. per sq. inch
□ across grain, 1000 lbs. per sq. inch
× with grain, 100 lbs. per sq. inch
○ across grain, 100 lbs. per sq. inch

Fig. 8. Position vs. Density for White Oak at Various lengths of soaking.



△ with grain, 1000 lbs. per sq. inch
□ across grain, 1000 lbs. per sq. inch
× with grain, 100 lbs. per sq. inch
○ across grain, 100 lbs. per sq. inch

Fig. 9. Position vs. Density for Cottonwood at Various lengths of soaking.

effects: 1, there was a noticeable vaporization of the resin as detected by the odor emitted during the curing as well as the decrease in weight; 2, exudation was considerable in some of the samples; 3, end and side effects may have been great enough to introduce slight errors because of a differential in pressure.

At the lower pressure of 100 lbs. per sq. inch the density in general was observed to increase as the edge was approached along the grain. This indicates that not so much vaporization occurred and is what would be expected to occur. The density across the grain either decreased with distance from the center or remained practically the same as the variation in density with the grain. This indicates in the white oak and to some extent in the red oak that the amount of resin permeating the wood is nearly the same across the grain as along the grain.

CONCLUSIONS

There is found to be a definite increase in density with the length of soak. This is what would be expected to happen; however, the length of soak which would be most economically feasible remains to be determined.

The density of the wood is found to be materially changed because the resin decreases the elasticity of the wood and increases the dimensional stability. The method of impregnating is such that no large pressure treating vessels are required for injecting the resin solution. The greatest density in compregnated woods is found in the wood of greatest original density and seems to indicate that the main function of the resin is not the addition of weight but the ability to bond the compressed fibers and keep them from retaining their original size.

Uniform density throughout large pieces of wood may be more nearly obtained than would be indicated from the experimental data because of the large end effect of the experimental samples. The density obtained will depend upon the pressure under which the resin is cured.

The variation in the experimental data resulted from the equipment used since it was impossible to accurately control pressure in the processing. Some of the samples were therefore compressed more than others before impregnating and during curing period.

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TESTING OF DIMETHYLOLUREA PLASTIC

LIST OF TABLES

LIST OF FIGURES

INTRODUCTION

The effect of water upon a dimethylolurea plastic is to be determined. This plastic is made by heating a mixture of 4 parts dimethylolurea and 5 parts wood shavings under pressure and at a temperature of about 145° C. The procedure for making the plastic is from a private communication of Dr. G.C.Williams. The stability of this plastic when immersed in water of various temperatures and the effect of exposure to salt water for prolonged periods will be determined.

EXPERIMENTAL

The test block from which the samples were taken measured $\frac{1}{2}$ x$\frac{1}{2}$ x 5 inches and the samples for testing were cut so that they approximated cubes. Six samples from various test blocks were taken to give a represenative analysis. The samples were dimensioned with a vernier micrometer and weighed on an analytical balance. One set of samples was unsurfaced, i.e., as prepared, while the other had been surfaced by sanding until the original glaze had disappeared. The samples were immersed in an erlenmeyer flask and kept at the desired temperature for a period of 24 hrs. after which they were blown dry, weighed, and dimensioned.

A second set of samples was immersed in a 4 % salt solution at room temperature and the same procedure followed in determining moisture and volume change. The temperature was controlled by placing a thermometer in the erlenmeyer flask and placing the entire assembly in an oven which had been regulated for the desired temperature. Equilibrium of the water with the oven temperature was obtained before placing the samples in the water.

TABLE I

Stability of Dimethylolurea plastic in Distilled water
At various temperatures

Temp. °F	Avg. Percentage Increase			
	Unsurfaced		Surfaced	
	Volume	Weight	Volume	Weight
68	5.47	7.22	6.25	7.62
100	12.01	13.40	11.69	14.15
125	15.18	17.92	17.41	17.40
150	29.80	22.95	35.00	23.00

TABLE II

Stability of Dimethylolurea plastic in 4% Salt solution
at room temperature.

Time of immersion (days)	Average Percentage Increase			
	Unsurfaced		Surfaced	
	Volume	Weight	Volume	Weight
3	5.45	6.90	7.45	7.68
9	10.57	11.25	11.65	12.25
12	11.99	12.77	13.66	13.29
19	14.18	14.16	15.63	14.49
22	15.02	14.26	16.69	14.69
30	16.55	14.30	17.06	14.31
Dry	1.03	-9.93	1.16	-9.32

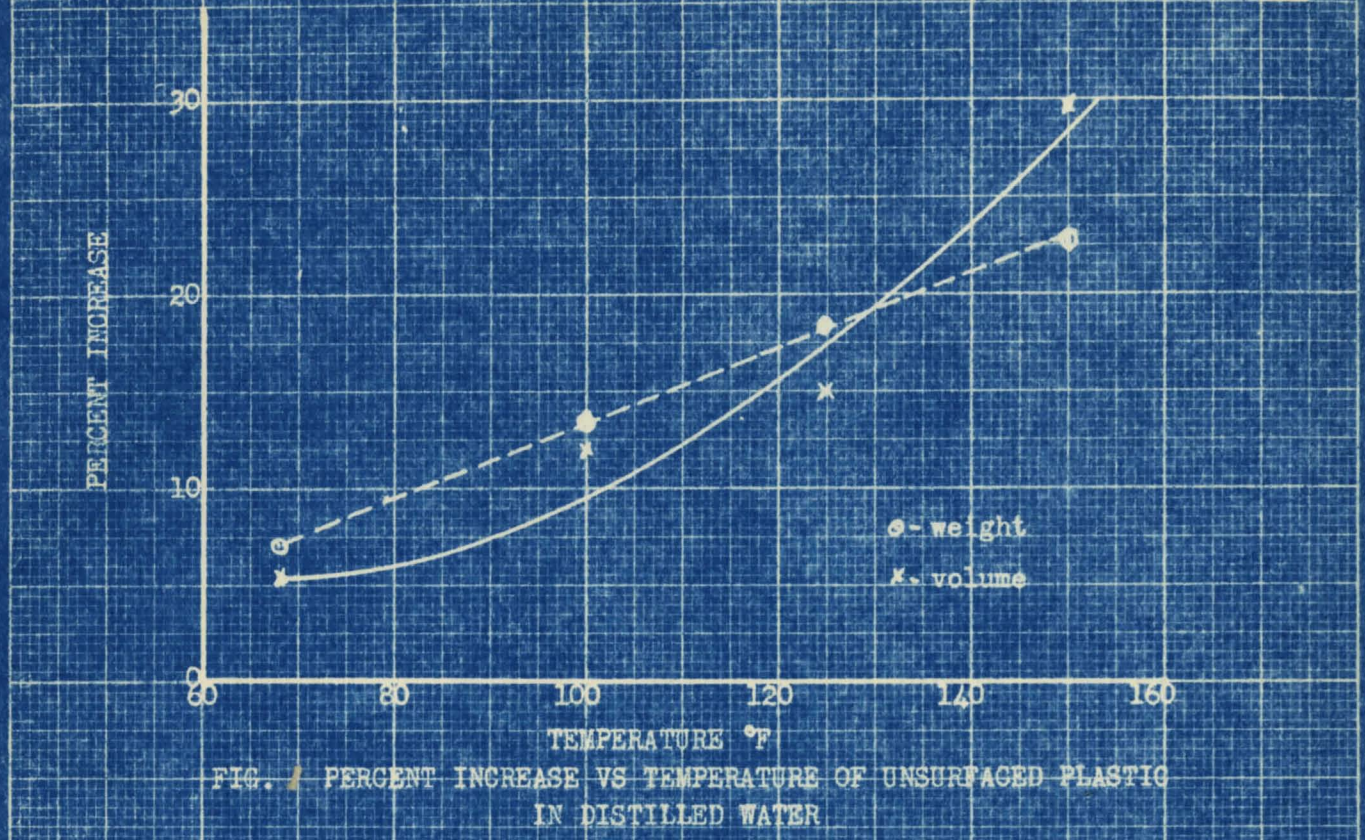

FIG. 1 PERCENT INCREASE VS TEMPERATURE OF UNSURFACED PLASTIC IN DISTILLED WATER

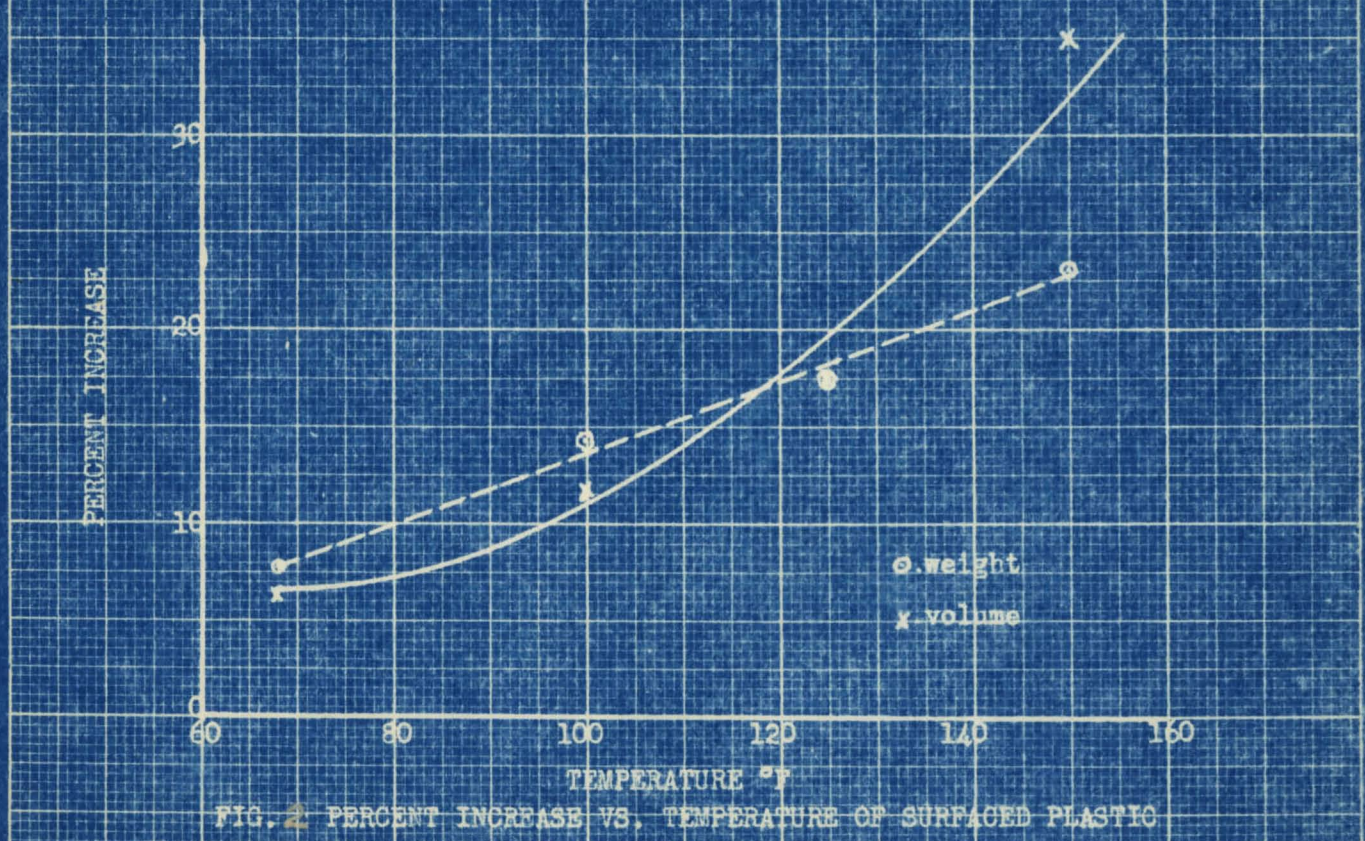

FIG. 2 PERCENT INCREASE VS. TEMPERATURE OF SURFACED PLASTIC IN DISTILLED WATER

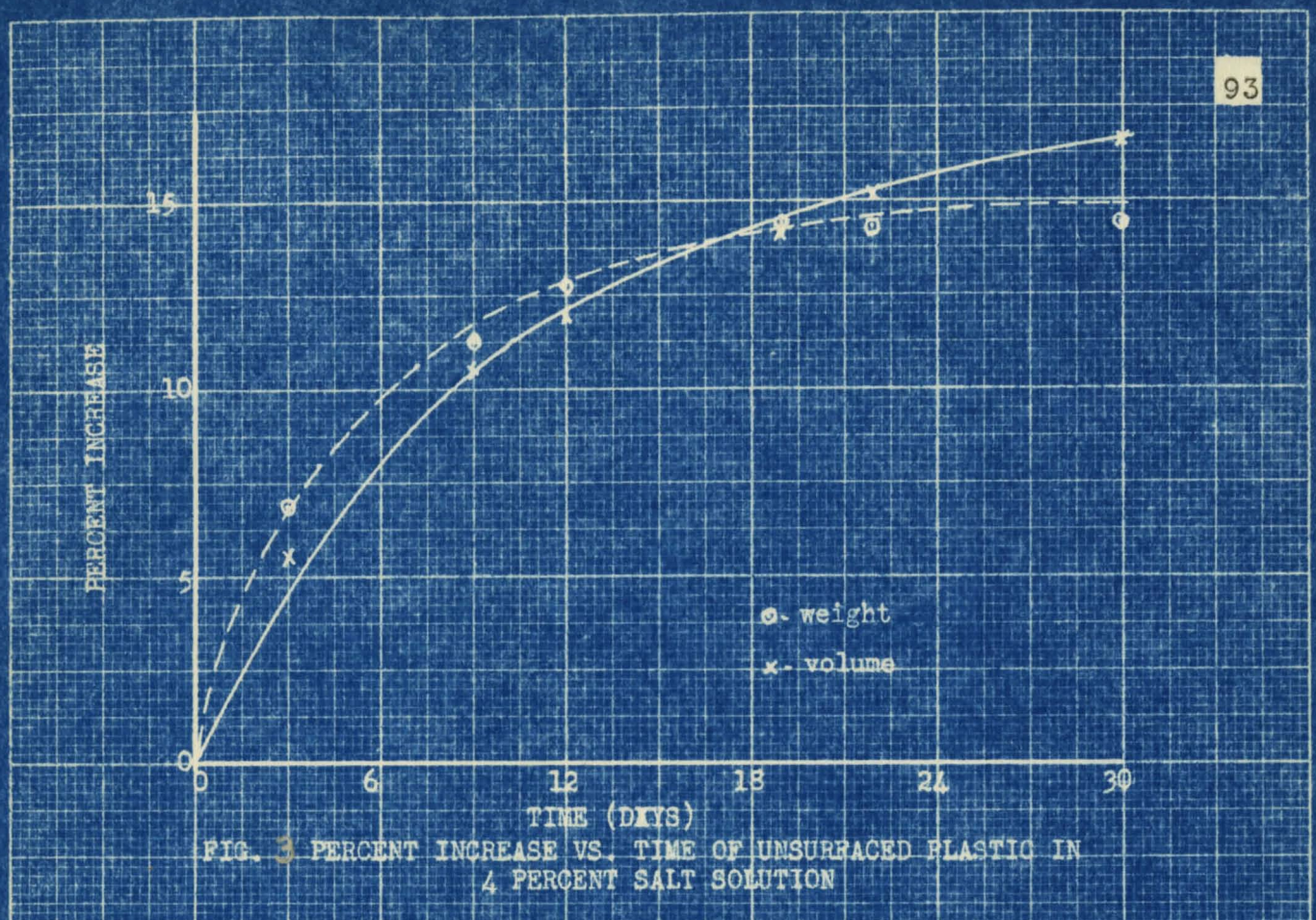

FIG. 3 PERCENT INCREASE VS. TIME OF UNSURFACED PLASTIC IN 4 PERCENT SALT SOLUTION

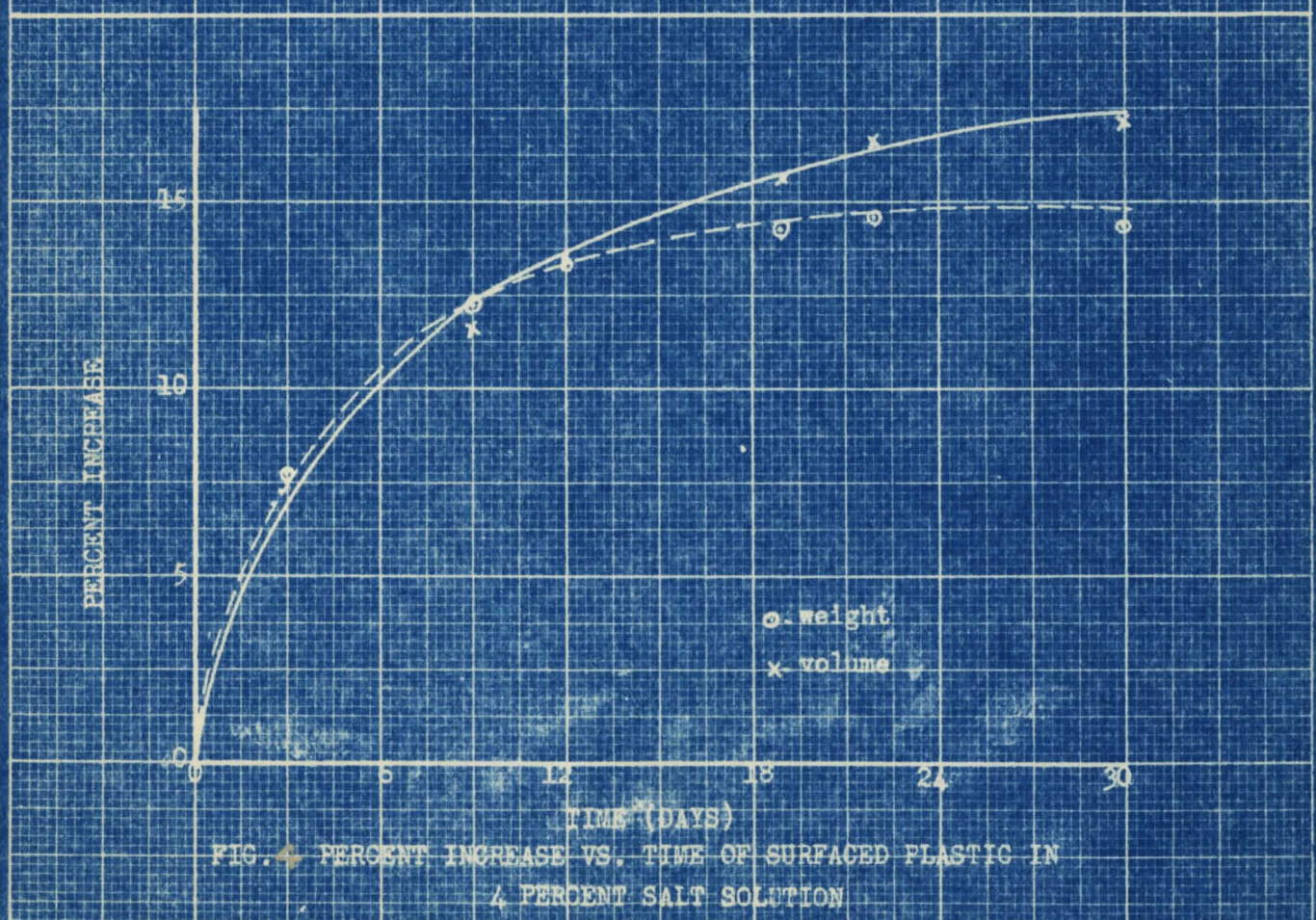

FIG. 4 PERCENT INCREASE VS. TIME OF SURFACED PLASTIC IN 4 PERCENT SALT SOLUTION

RESULTS AND CONCLUSIONS

From the data presented in Tables I and II and from Figures 1 to 4 the following conclusions and results are arrived at.

This plastic has no value when exposed to water at elevated temperatures as it rapidly deterioates. The samples were observed to break into small pieces at the temperature of boiling water. At lower temperatures there was a definite swelling of the plastic. The degree of swelling increased with increasing temperature of the water. The swelling was observed to be greatest in the direction of compression.

The swelling is rapid at the beginning of immersion in salt water but later decreases. The weight increase tends to approach a constant value. From the data it is seen that upon drying, the original volume is again closely attained. The dimension change of the samples was noticeable, and a slight swelling can be detected. The weight loss is considerable and indicates a solubility of the plastic. There is no appreciable difference between the behavior of the surfaced and unsurfaced examples in either test.

ACKNOWLEDGMENTS

The author wishes to thank Gamble Bros., whose division of Industrial Research Fellowship made this investigation possible. He also wants to express his appreciation to Mr. C. D. Dosker who supervised this problem and to Mr. Charles F. Sacra who assisted in the experimental work.

VITA

James Irwin Stevens was born at Valley Station, Kentucky, on July 15, 1920. He received his primary and secondary education in the public schools of Jefferson County. He entered the Speed Scientific School of the University of Louisville in September 1938 and obtained the degree of Bachelor of Chemical Engineering in May, 1942. He received the degree of Master of Chemical Engineering in May, 1943.

Mr. Stevens was awarded the Federation of Paint and Varnish Productions Men's Club Fellowship as well as a D.I.R. fellowship at the University of Louisville for the year 1942-1943. Upon completion of this work he was employed by the University of Louisville as an Instructor in Chemical Engineering.

He is a member of Sigma Tau, National Honorary Engineering Fraternity, and of Theta Chi Delta, National Honorary Chemistry Fraternity.